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The Crystal Engineering of Radiation-Sensitive Diacetylene Cocrystals and Salts

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Supplementary Information

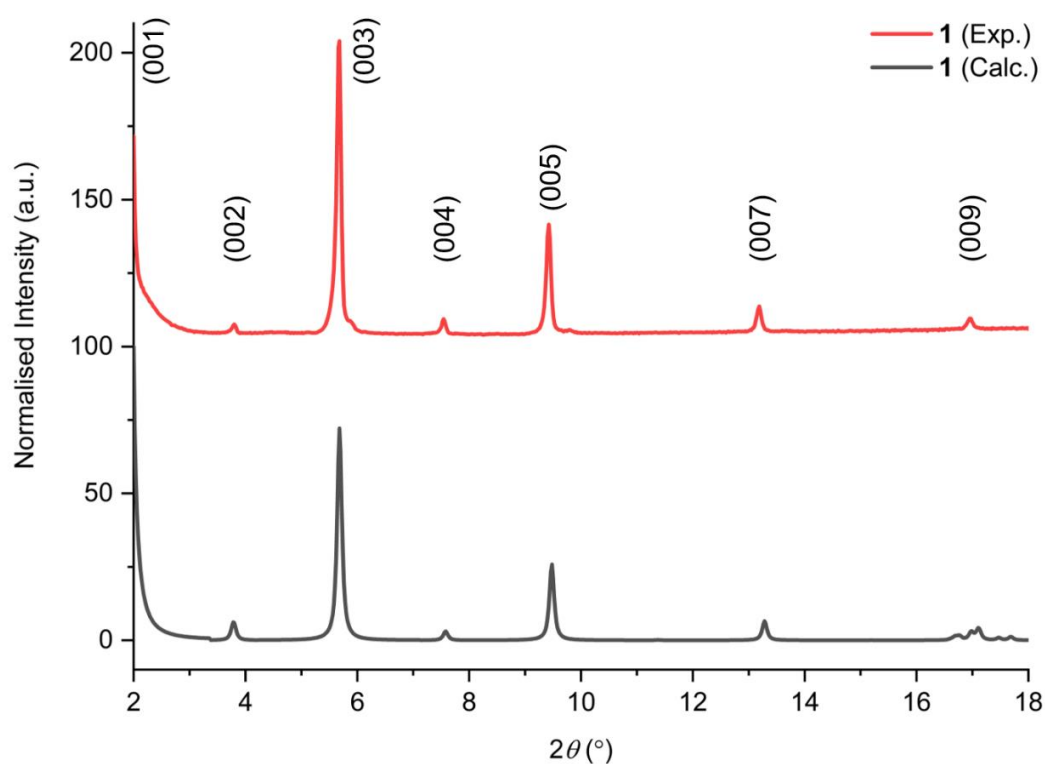


Figure S1. The experimental and calculated PXRD patterns of **1** highlighting the initial (00l) reflections. The experimental data were collected at room temperature (approx. 293 K), with the calculated data collected at 100 K.



Figure S2. A photograph of **1** before any irradiation studies. Highlighting that the surface colouration of the crystals is not representative of the bulk material (no evidence of polymerisation).

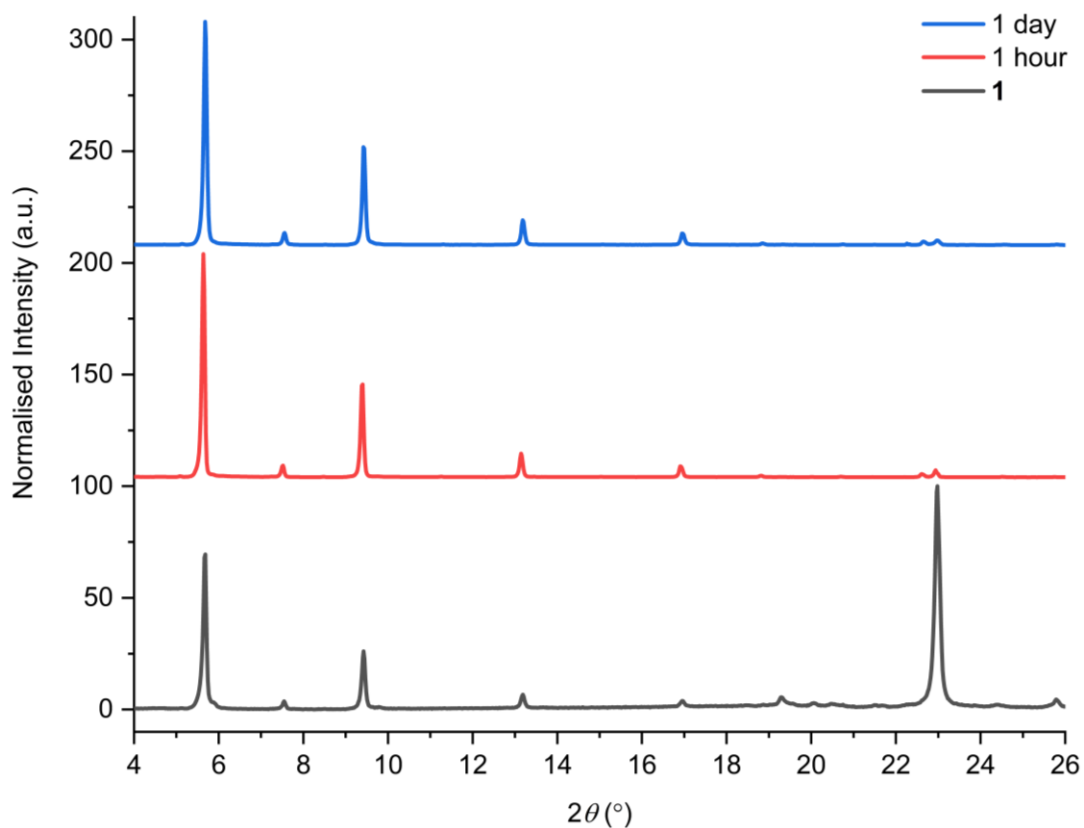


Figure S3. The experimental PXRD patterns of **1** irradiated for one hour and one day. The relative intensities are affected by differences in preferred orientation between the irradiated and non-irradiated samples.

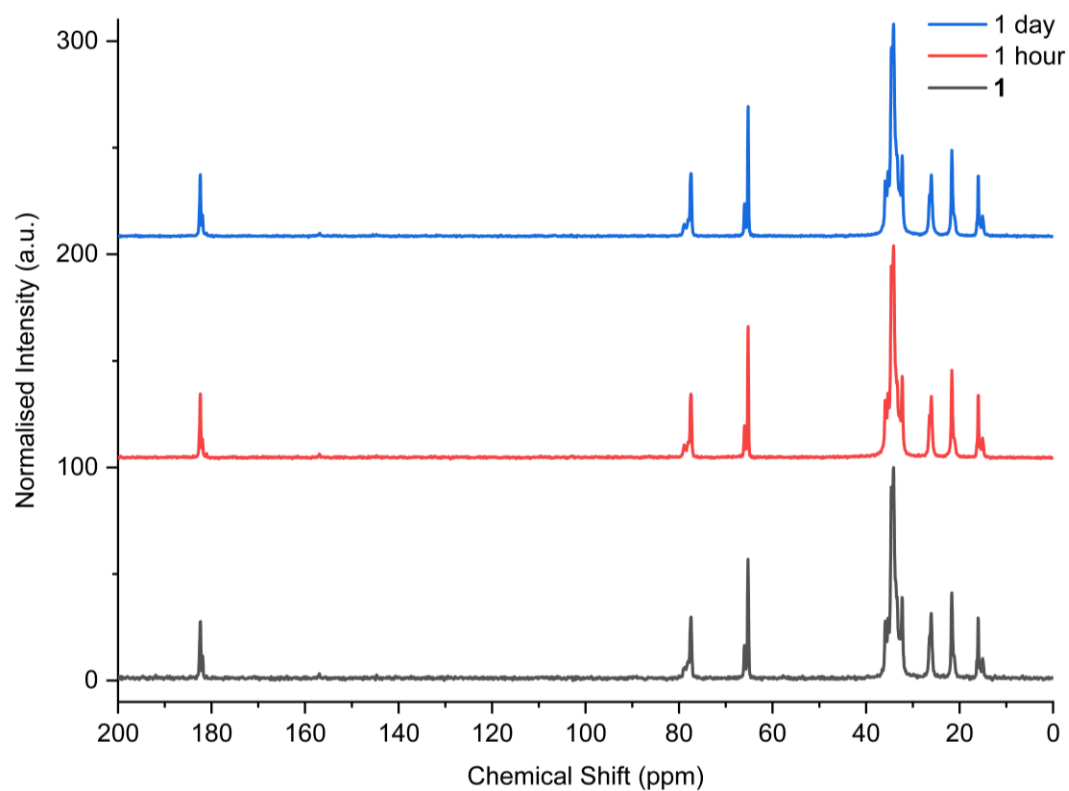


Figure S4. CP-MAS ^{13}C NMR spectra of **1** irradiated for different durations by UV light at 254 nm.

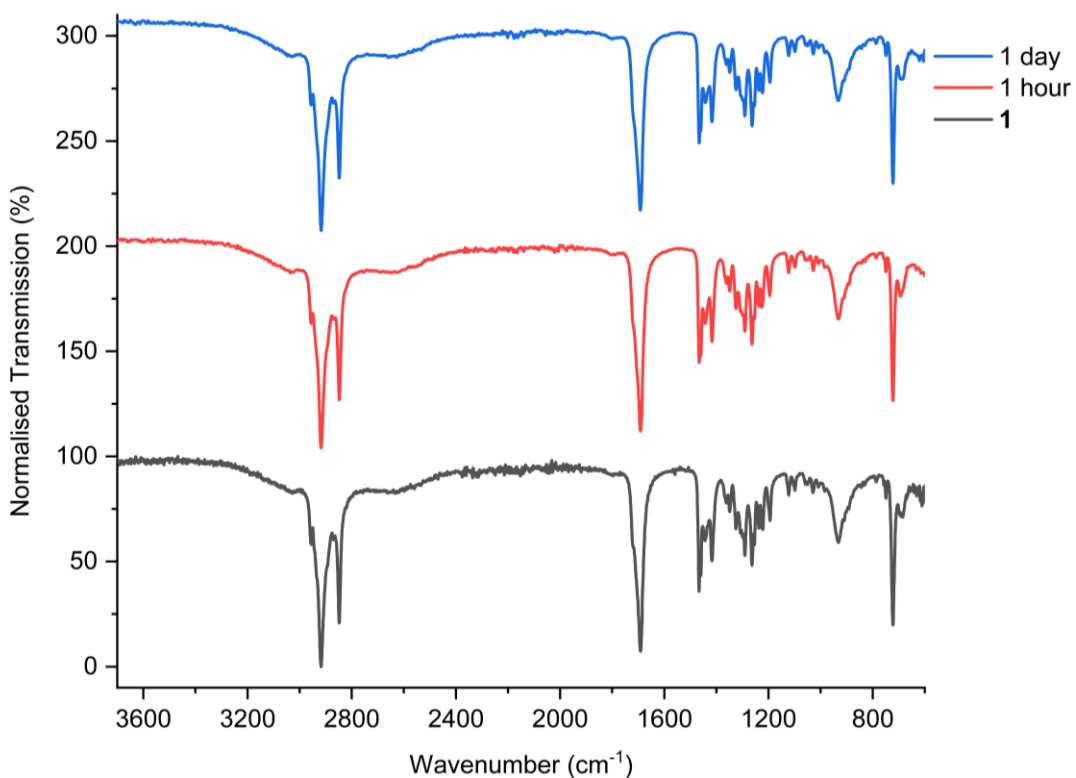


Figure S5. The FTIR spectra of **1** irradiated for different durations by UV light at 254 nm.

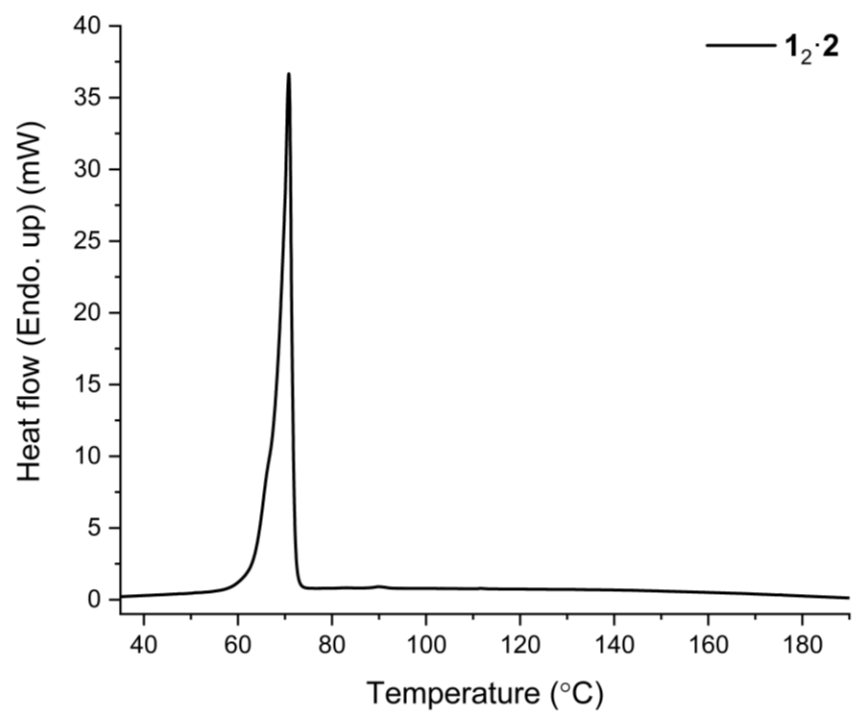


Figure S6. The DSC thermogram of **1₂·2** with a peak onset endotherm at 56.9 °C.

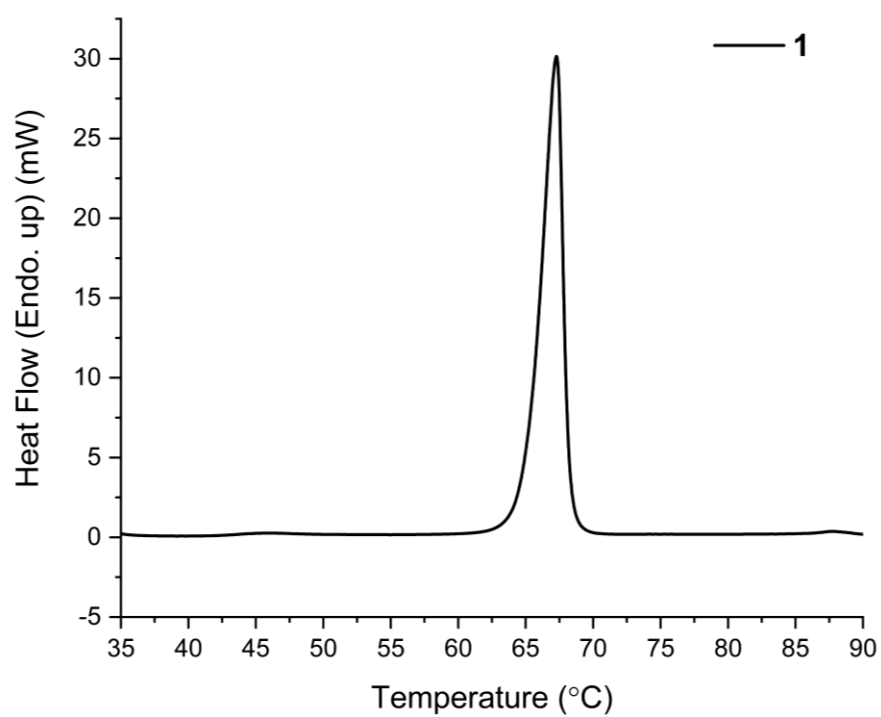


Figure S7. The DSC thermogram of **1** with a melt onset endotherm at 62.3 °C.

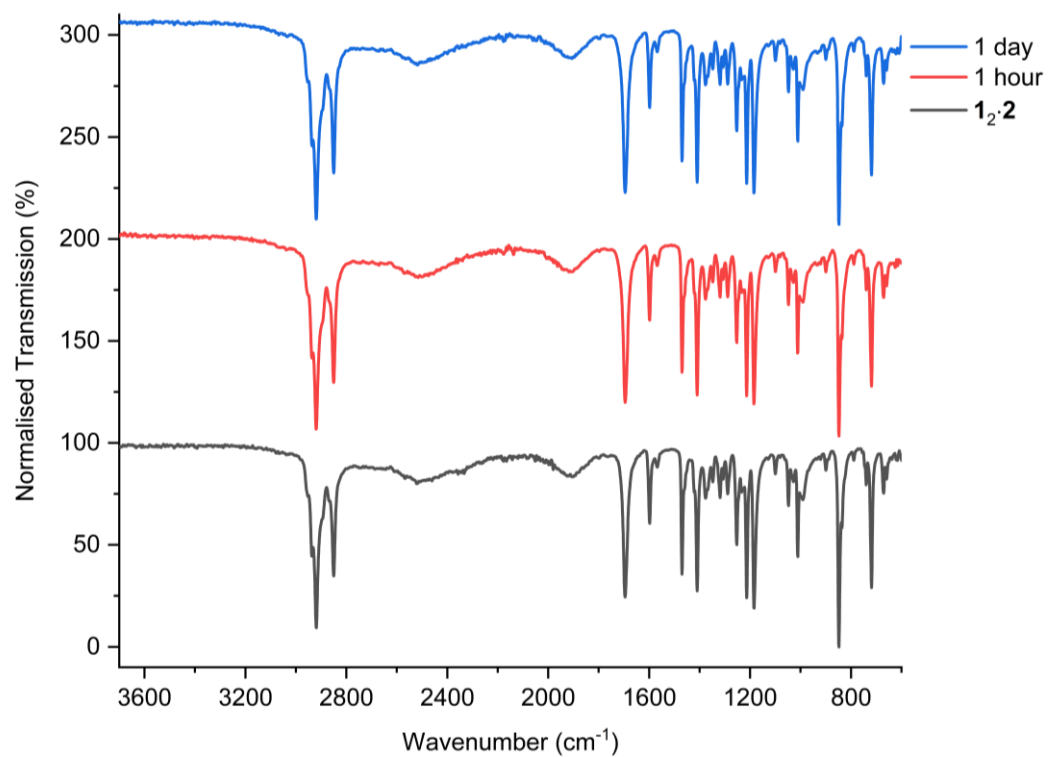


Figure S8. The FTIR spectra of **1₂·2** irradiated for one hour by UV light at 365 nm.

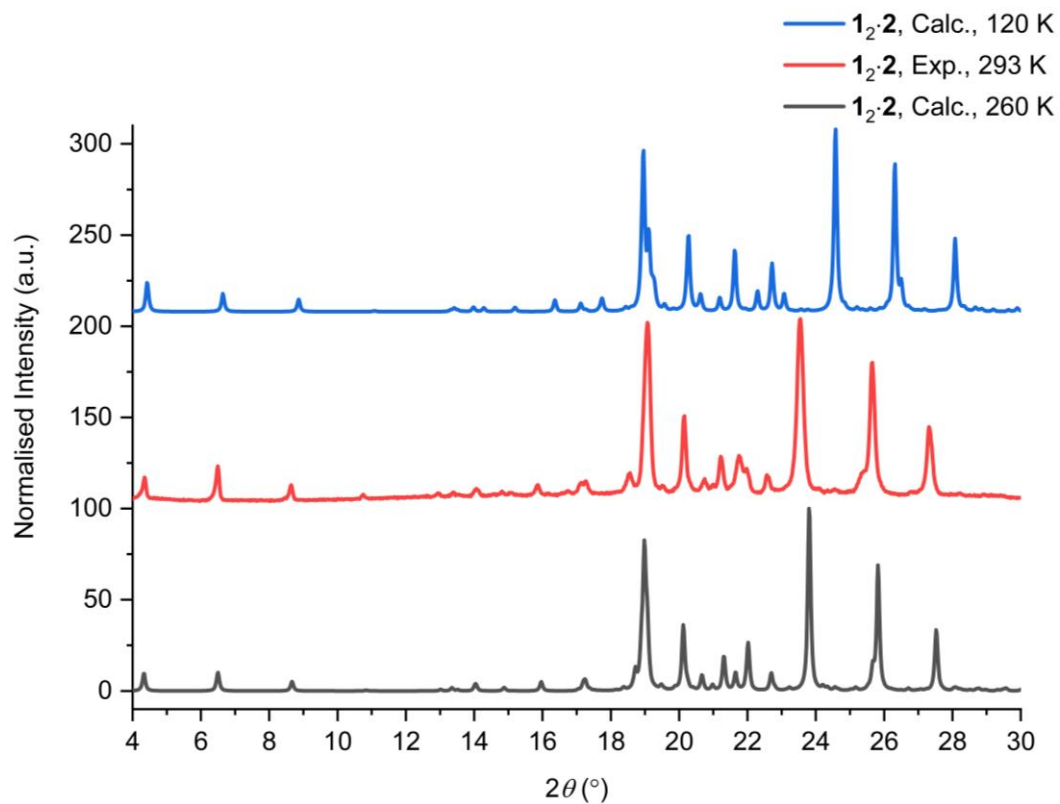


Figure S9. The calculated and experimental PXRD pattern of **1₂·2** at different temperatures.

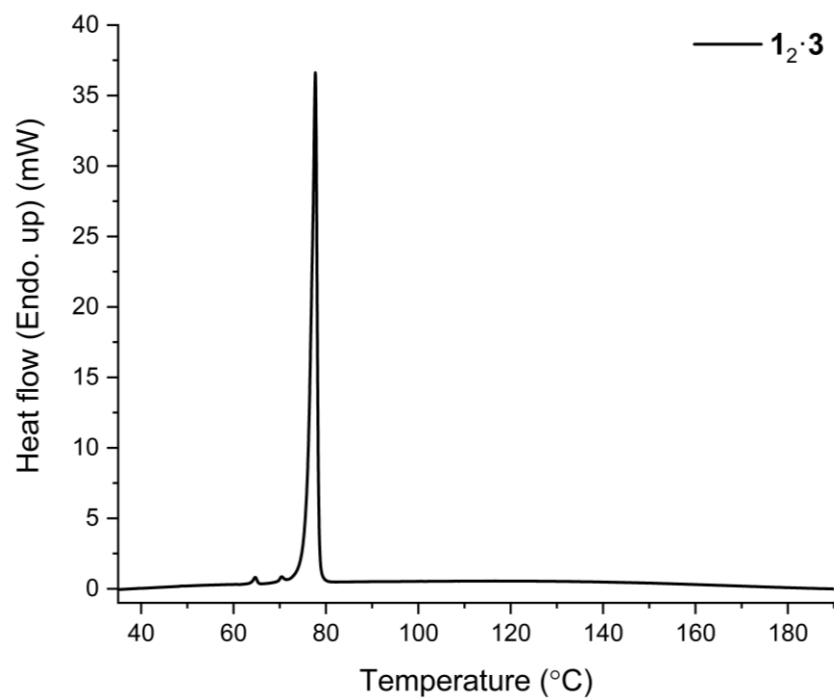


Figure S10. The DSC thermogram of $1_2 \cdot 3$ with a melt onset endotherm at 73.4 °C, with a residual peak of **1** at 63.7 °C.

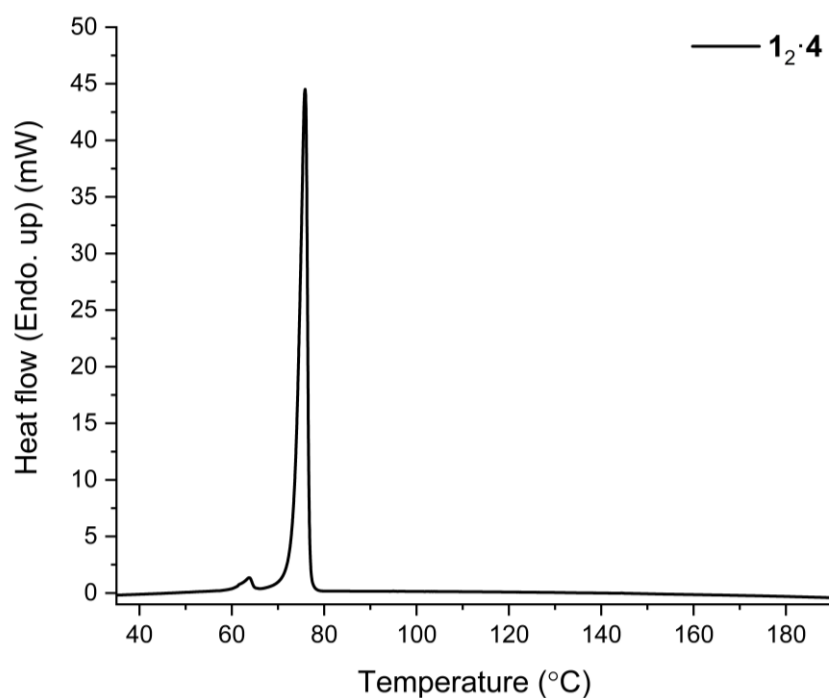


Figure S11. The DSC thermogram of $1_2 \cdot 4$ displaying a melt onset endotherm at 72.2 °C, with residual unreacted **1** at 62.6 °C.

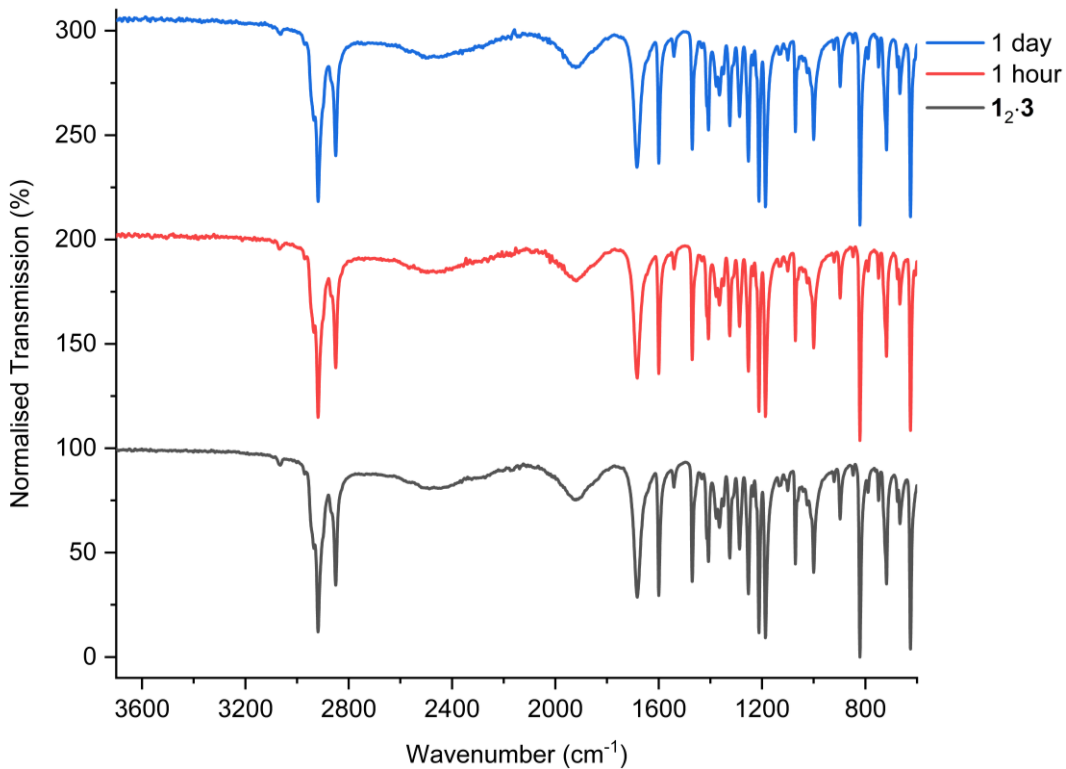


Figure S12. The FTIR spectra of $1_2 \cdot 3$ irradiated for different durations by UV light at 254 nm.

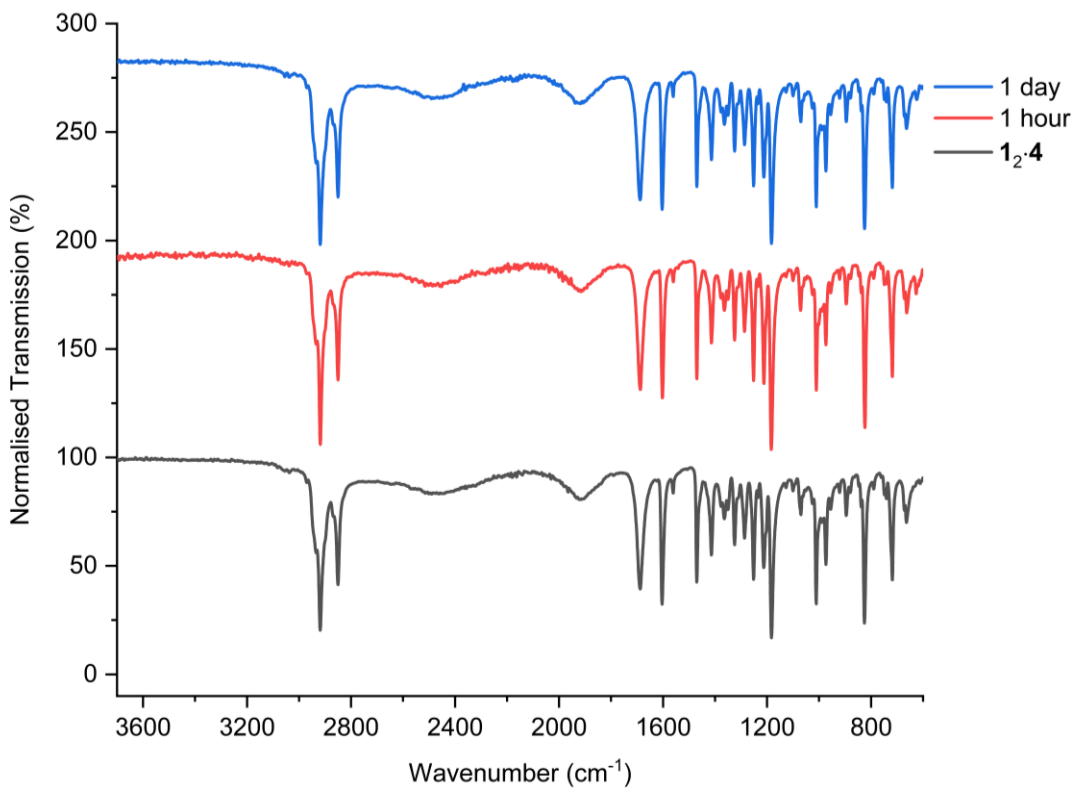


Figure S13. The FTIR spectra of $1_2 \cdot 4$ irradiated for different durations by UV light at 254 nm.

Cocrystal	Temp.	a / Å	b / Å	c / Å	β / °	Volume / Å ³
1₂·3	100 K	5.4415(2)	8.9535(4)	55.673(3)	90.8823(10)	2712.1(2)
	260 K	5.4783(3)	8.9976(5)	56.951(4)	92.506(2)	2804.5(3)
	273 K	5.4761(3)	8.9950(5)	57.173(4)	92.710(2)	2813.0(5)
1₂·4	120 K	5.4494(3)	8.9235(5)	57.441(3)	92.643(2)	2790.2(3)
	260 K	5.4847(4)	8.9801(6)	58.562(4)	90.849(3)	2884.1(3)
	273 K	5.503(5)	9.023(7)	58.93(5)	90.24(3)	2927.0(7)

Table S1. The unit cell axes of **1₂·3** and **1₂·4** collected at various temperatures.

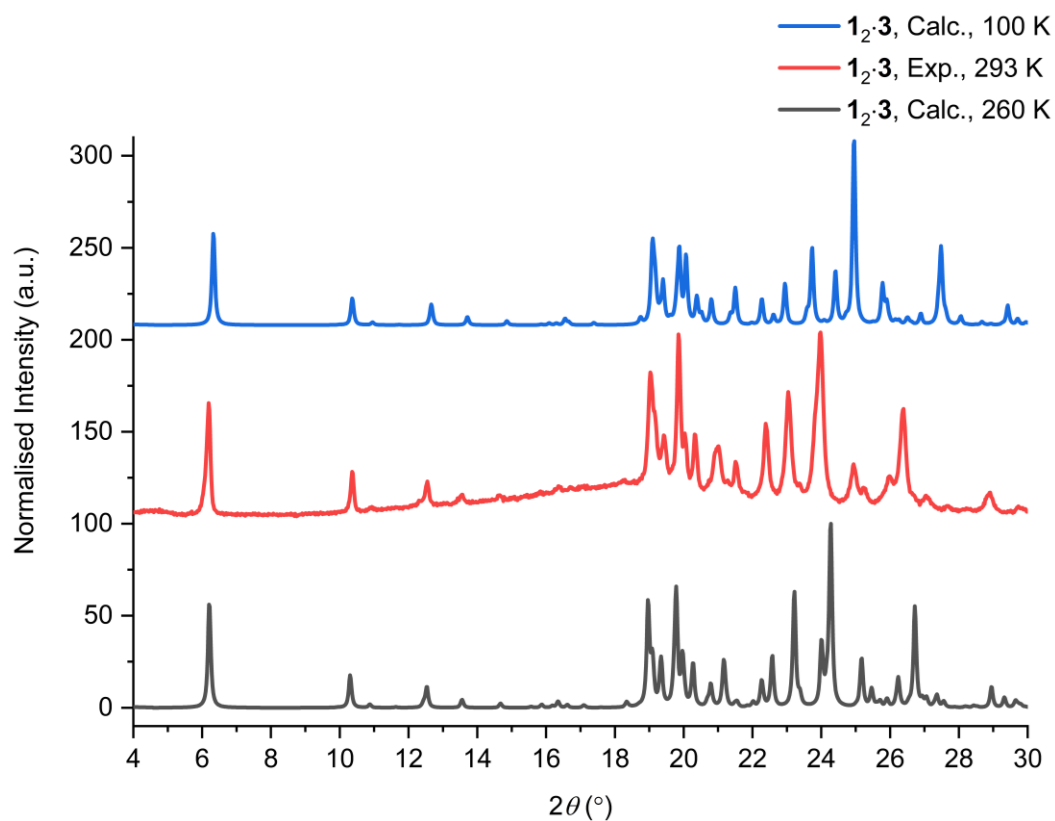


Figure S14. The calculated and experimental PXRD patterns of **1₂·3** at different temperatures.

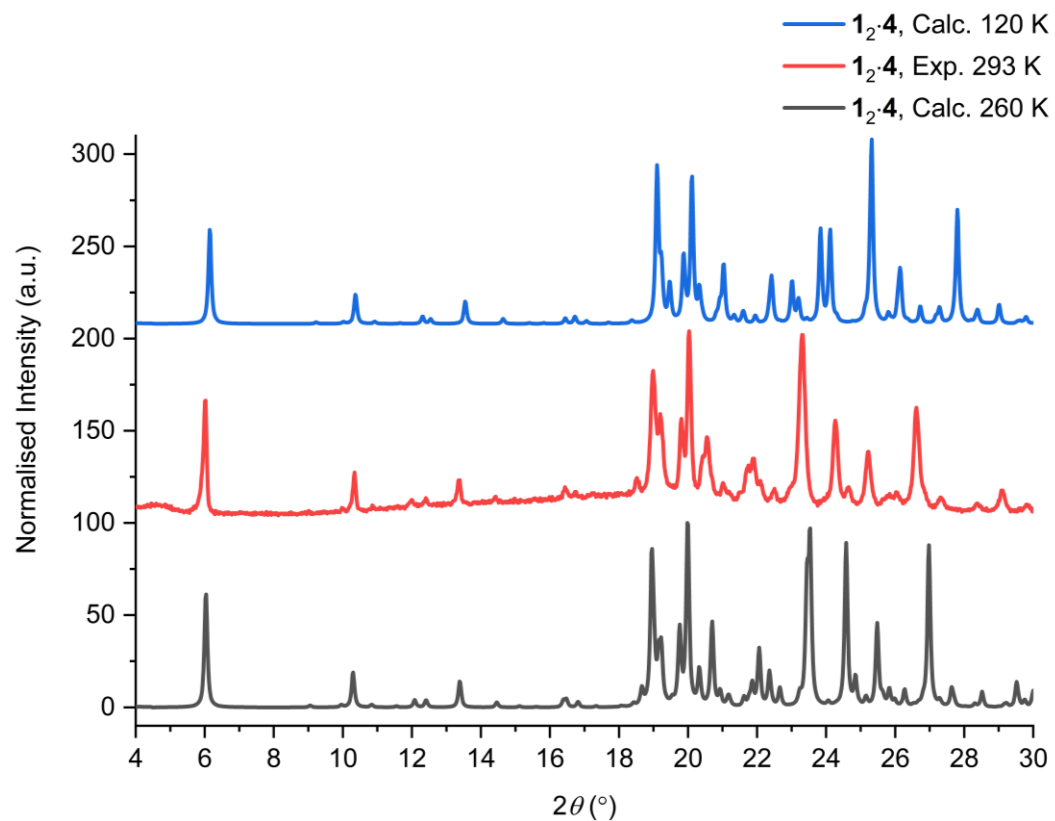


Figure S15. The calculated and experimental PXRD patterns of $1_2 \cdot 4$ collected at different temperatures.

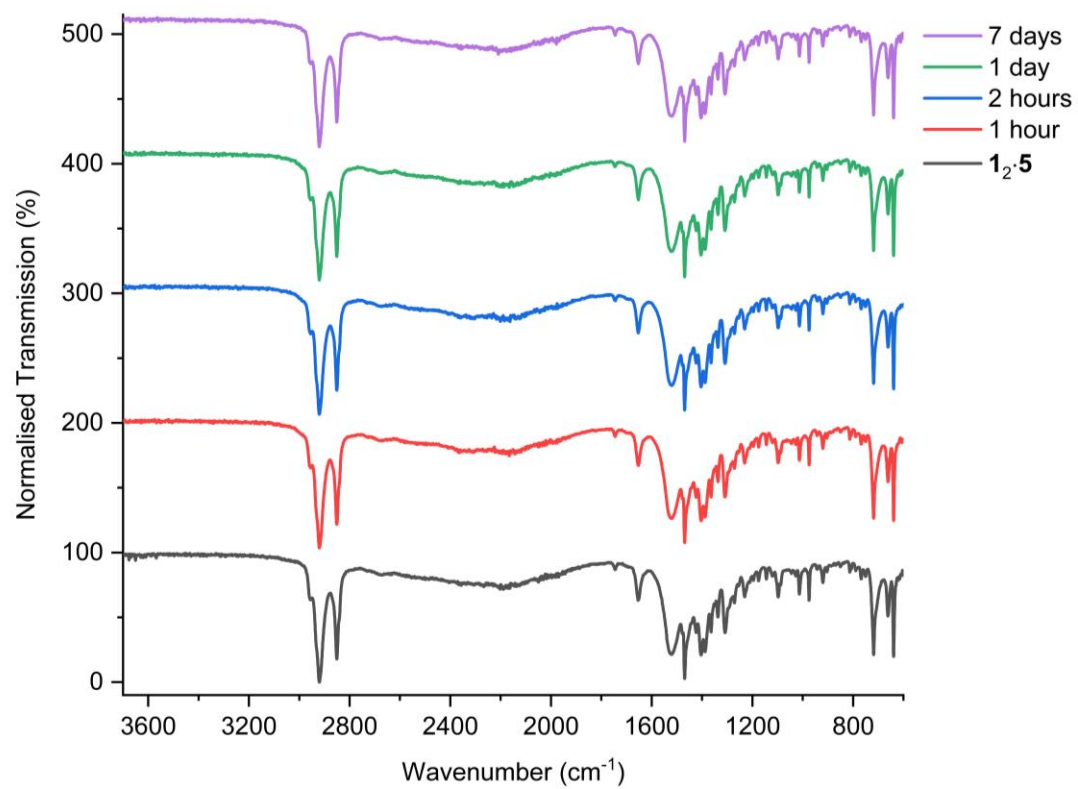


Figure S16. The FTIR spectra of $1_2 \cdot 5$ irradiated for different durations by UV light at 254 nm.

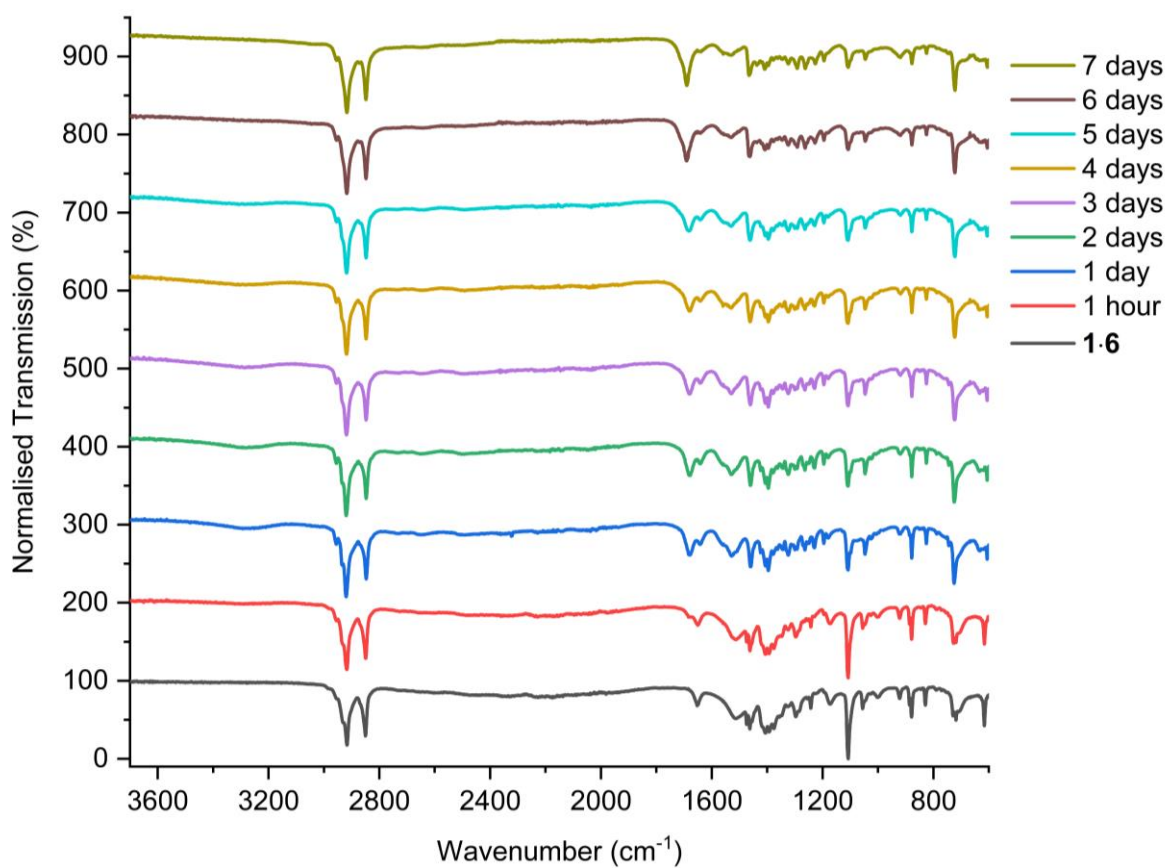


Figure S17. The FTIR spectra of **1·6** irradiated for different durations by UV light at 254 nm.

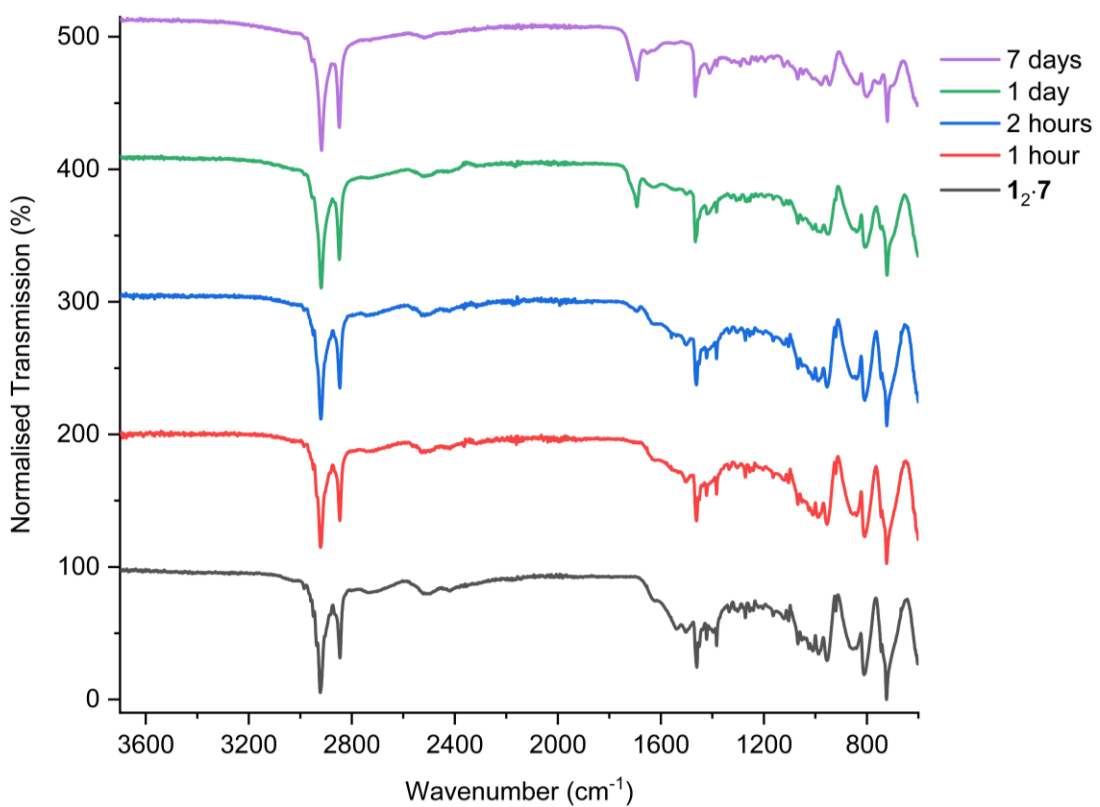


Figure S18. The FTIR spectra of $1_2 \cdot 7$ irradiated for different durations by UV light at 254 nm.

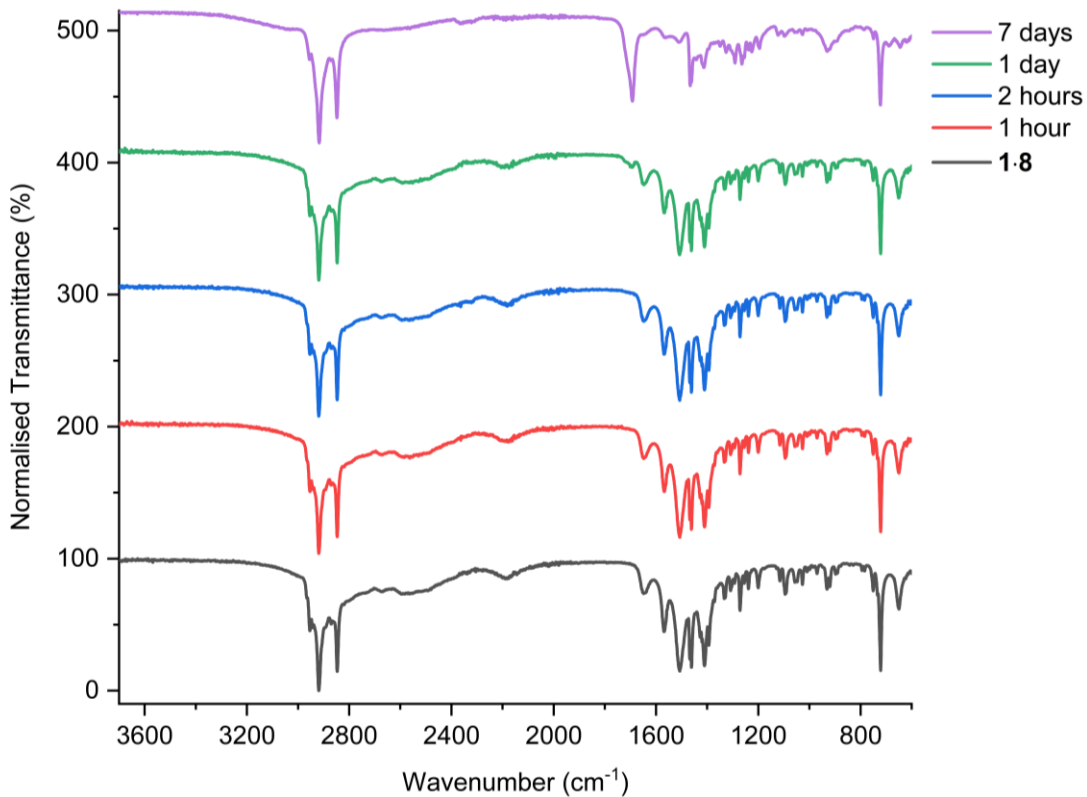


Figure S19. The FTIR spectra of **1·8** irradiated for different durations by UV light at 254 nm.

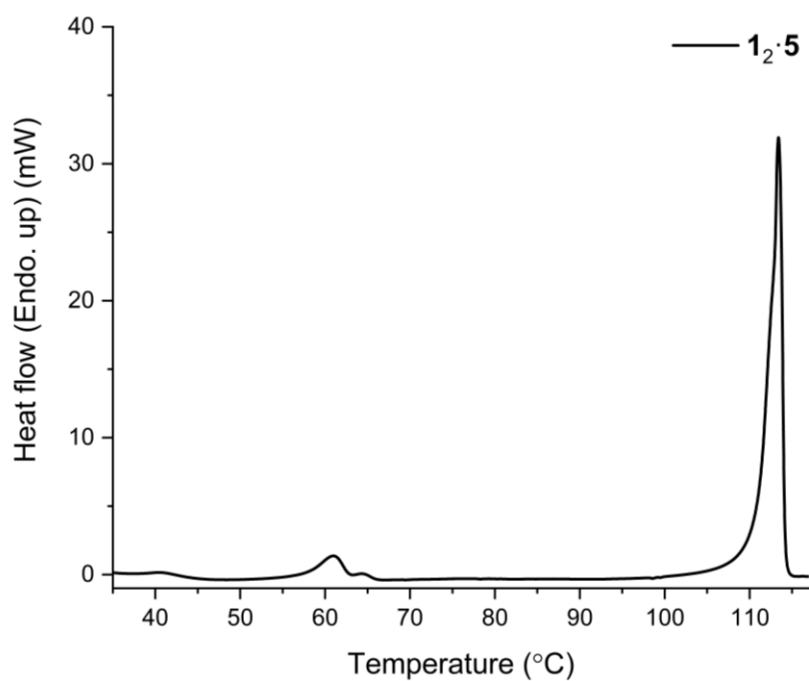


Figure S20. The DSC thermogram of **1₂·5** displaying a melt onset endotherm at 107.4 °C with residual unreacted **1** between 59.5-64.6 °C.

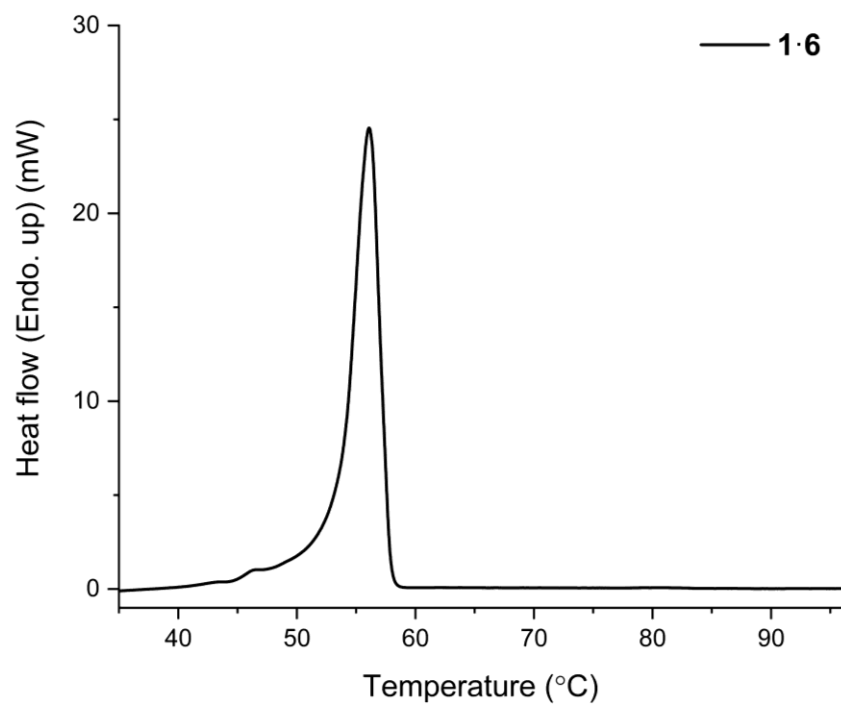


Figure S21. The DSC thermogram of **1·6** displaying a melt onset endotherm at 43.8 °C.

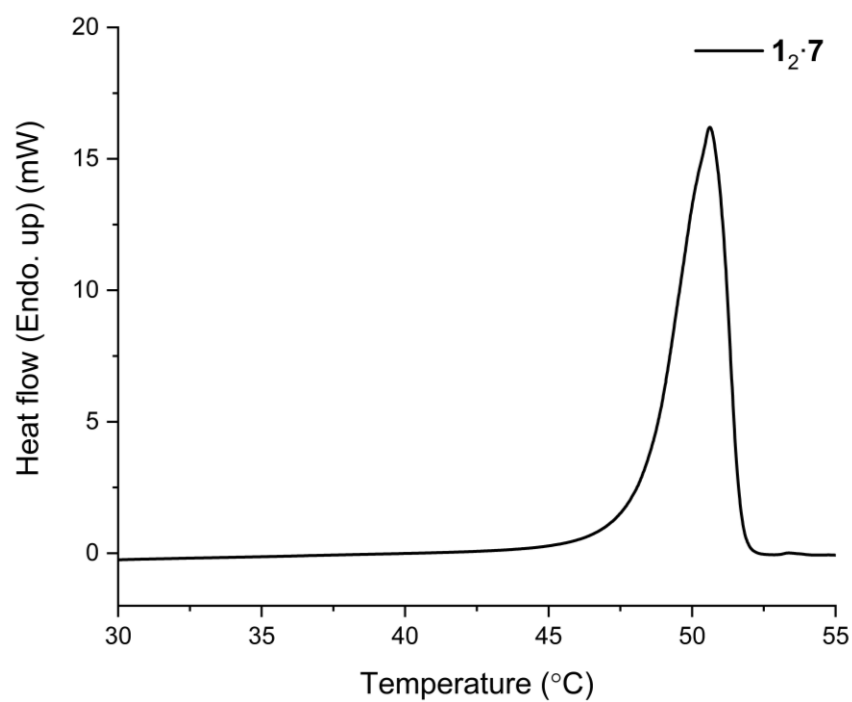


Figure S22. The DSC thermogram of **1₂·7** displaying a melt onset endotherm at 45.8 °C.

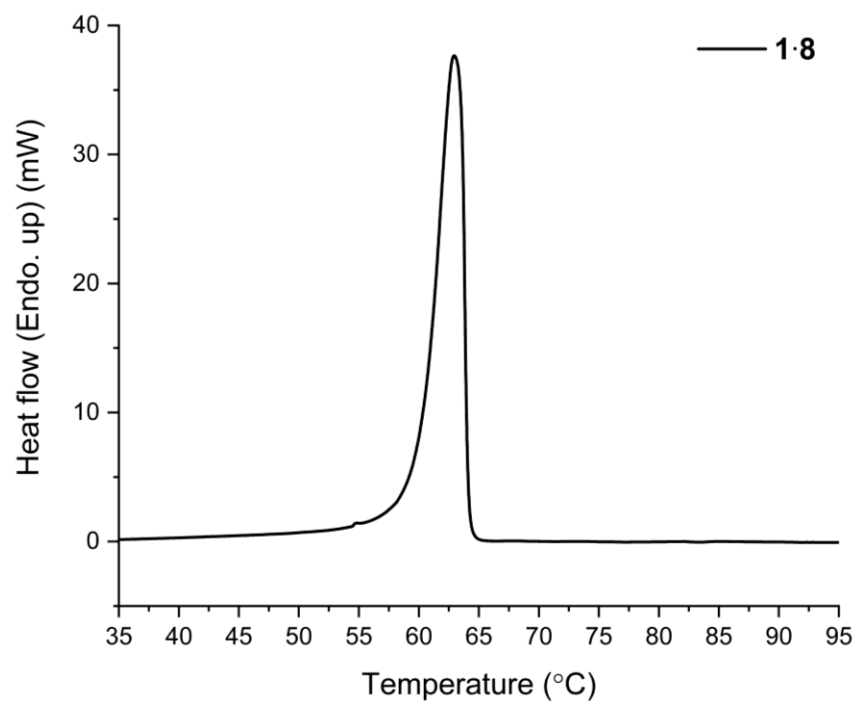


Figure S23. The DSC thermogram of **1·8** displaying a melt onset endotherm at 57.3 °C.

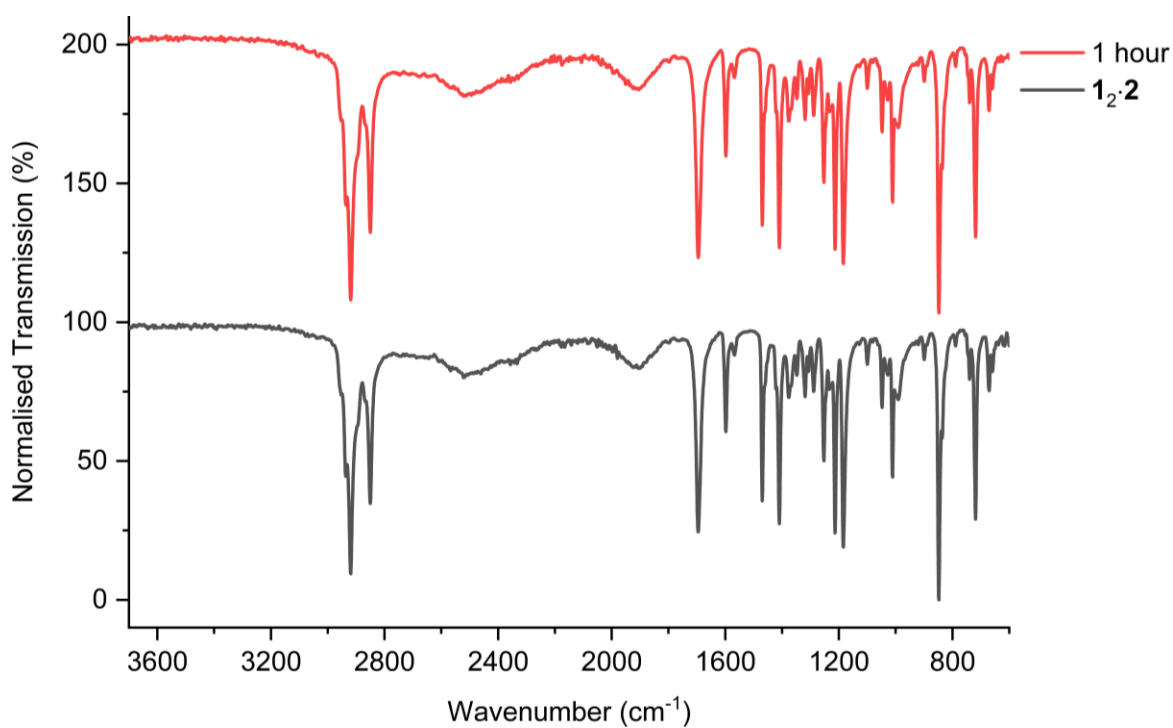


Figure S24. The FTIR spectra of **1₂·2** irradiated for one hour by UV light at 254 nm.

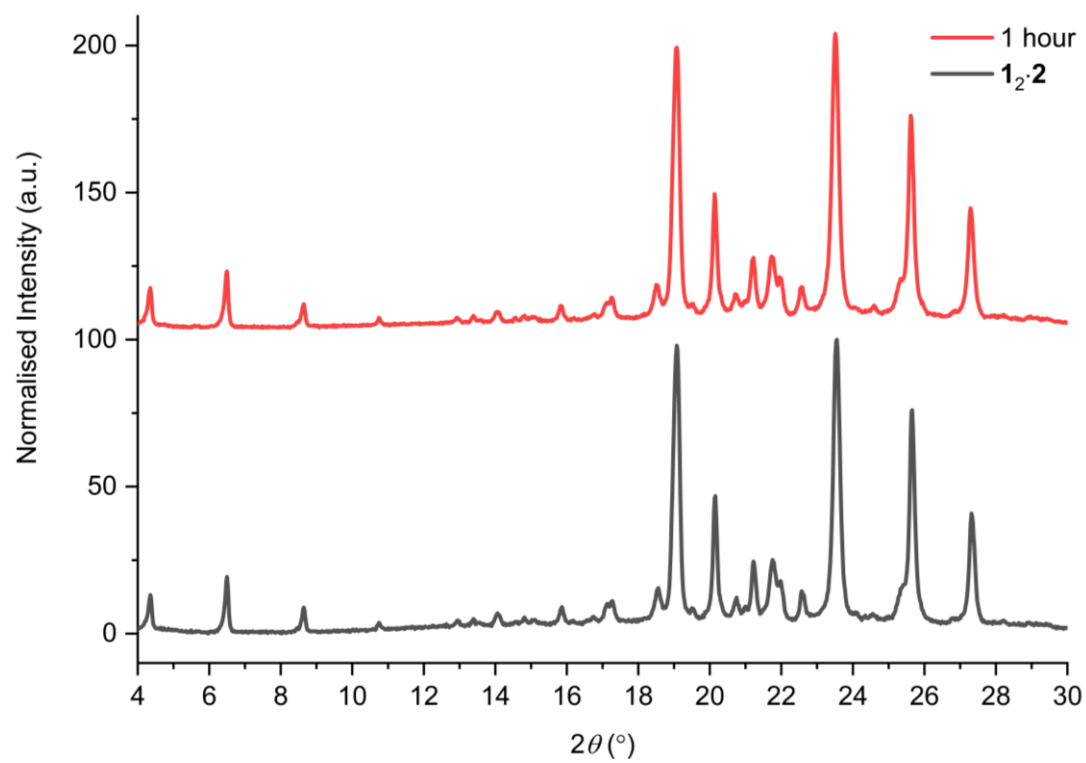


Figure S25. The experimental PXRD patterns of $1_2 \cdot 2$ irradiated for one hour by UV light at 254 nm.

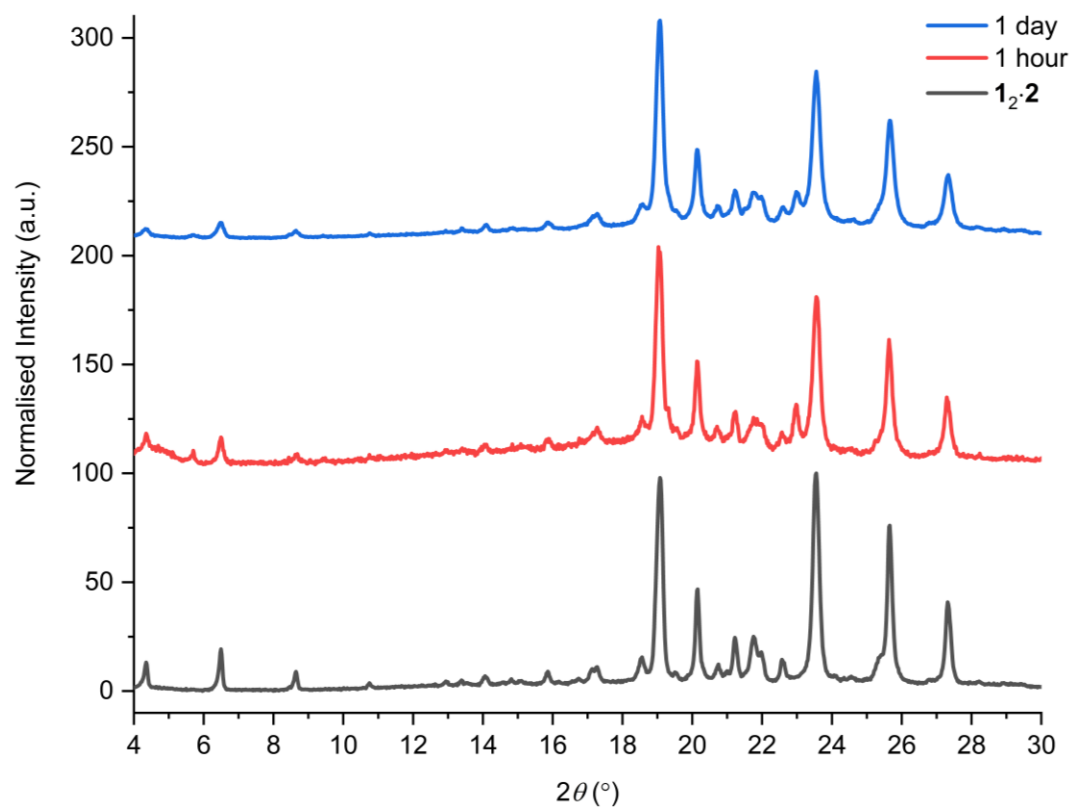


Figure S26. The experimental PXRD patterns of $1_2 \cdot 2$ irradiated for different durations by UV light at 365 nm. Additional peak at $23.0^\circ 2\theta$ in the one hour and one day patterns correlate to **1**.

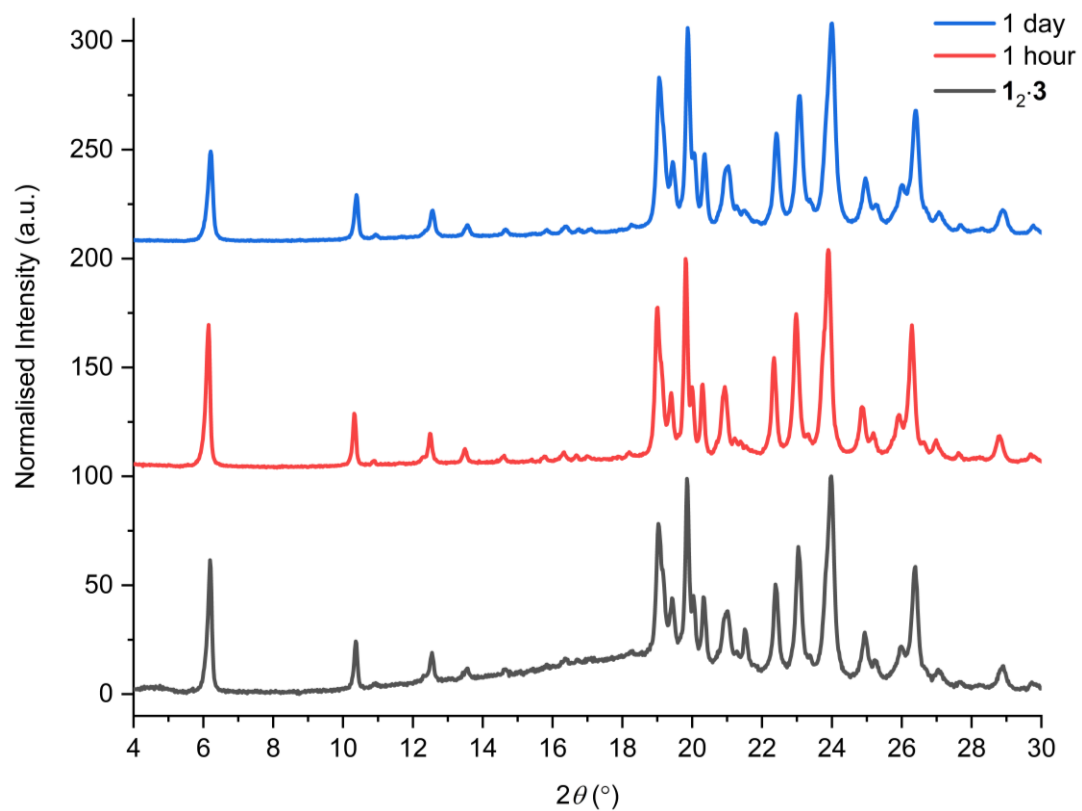


Figure S27. The experimental PXRD patterns of $1_2 \cdot 3$ irradiated for different durations by UV light at 254 nm.

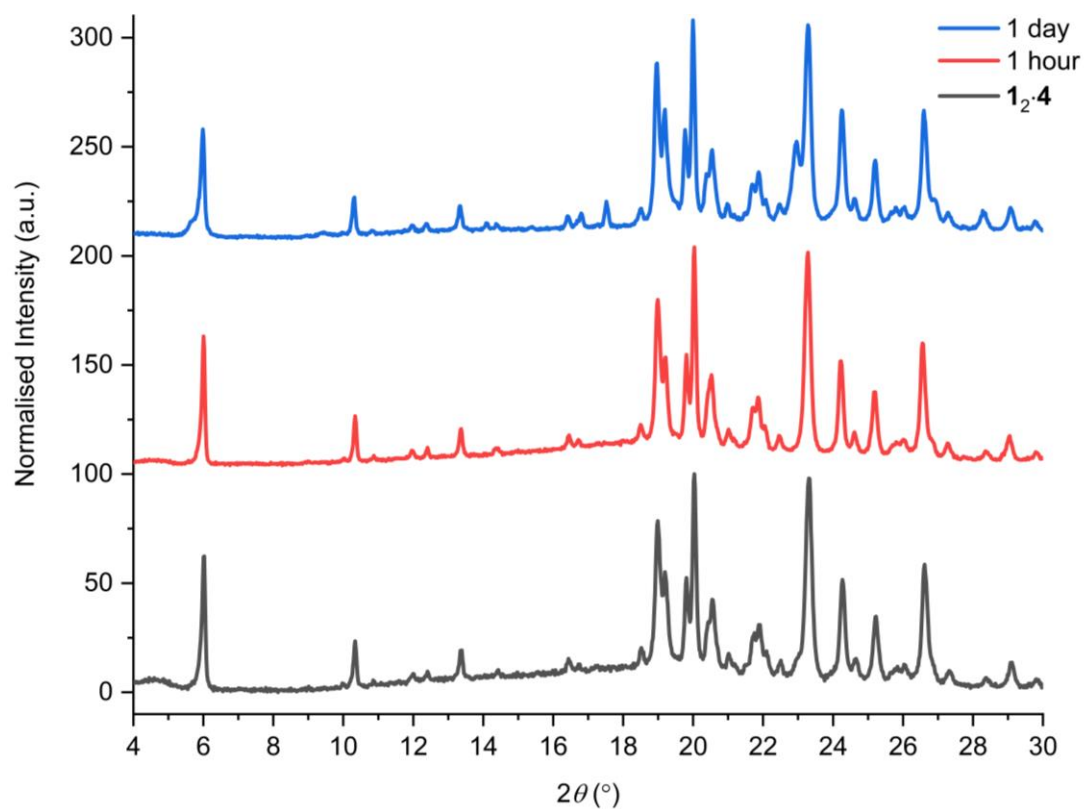


Figure S28. The experimental PXRD patterns of **1₂·4** irradiated for different durations by UV light at 254 nm. The peaks emerging in the one-day irradiated patterns at 5.7, 17.5, and 23.0 2θ , correlate to peaks of **1**.

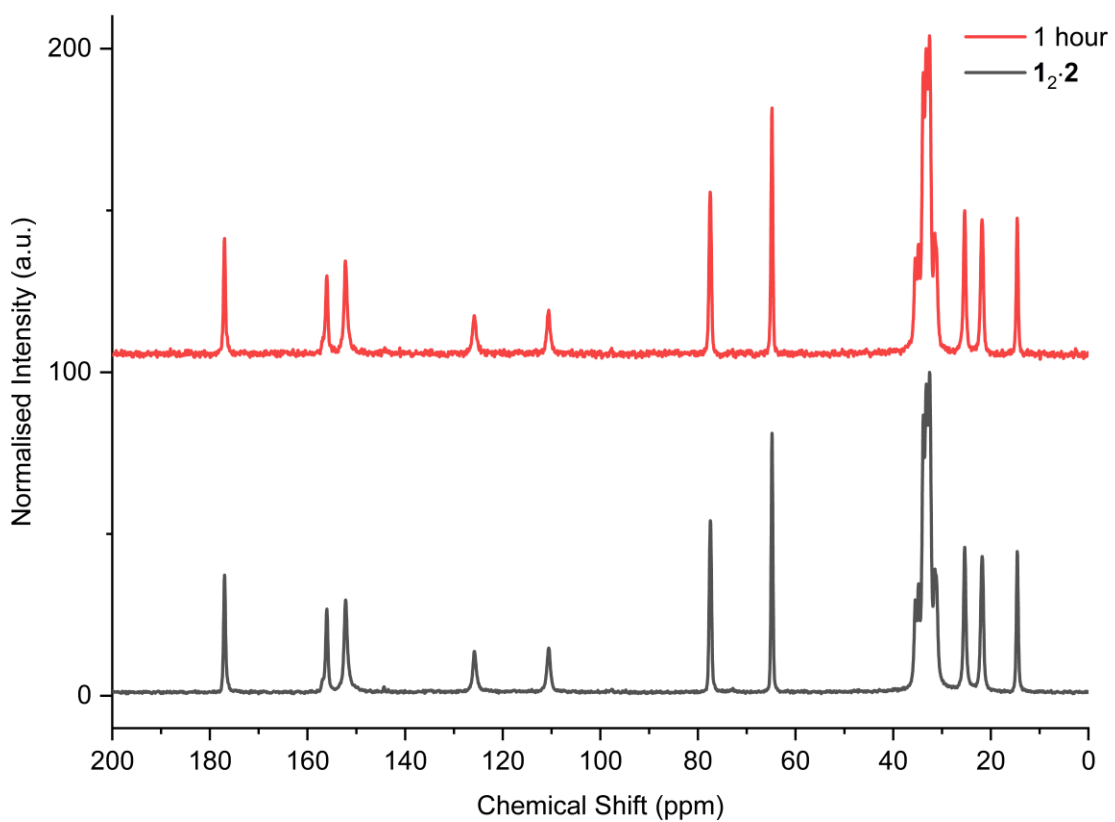


Figure S29. CP-MAS ^{13}C NMR spectra of $1_2 \cdot 2$ irradiated for different durations by UV light at 254 nm.

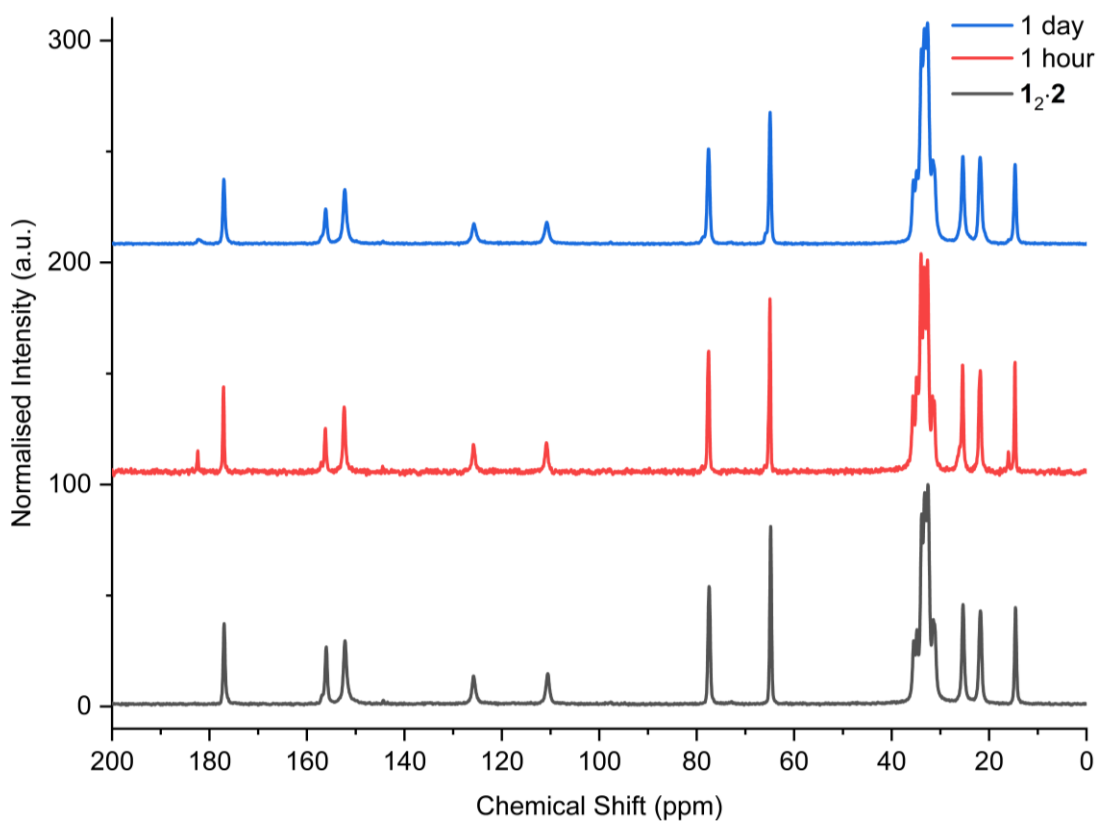


Figure S30. CP-MAS ^{13}C NMR spectra of $1_2\cdot 2$ irradiated for one hour by UV light at 365 nm. The additional peak at 182.5 ppm which correlates to the carboxylate peak of **1**.

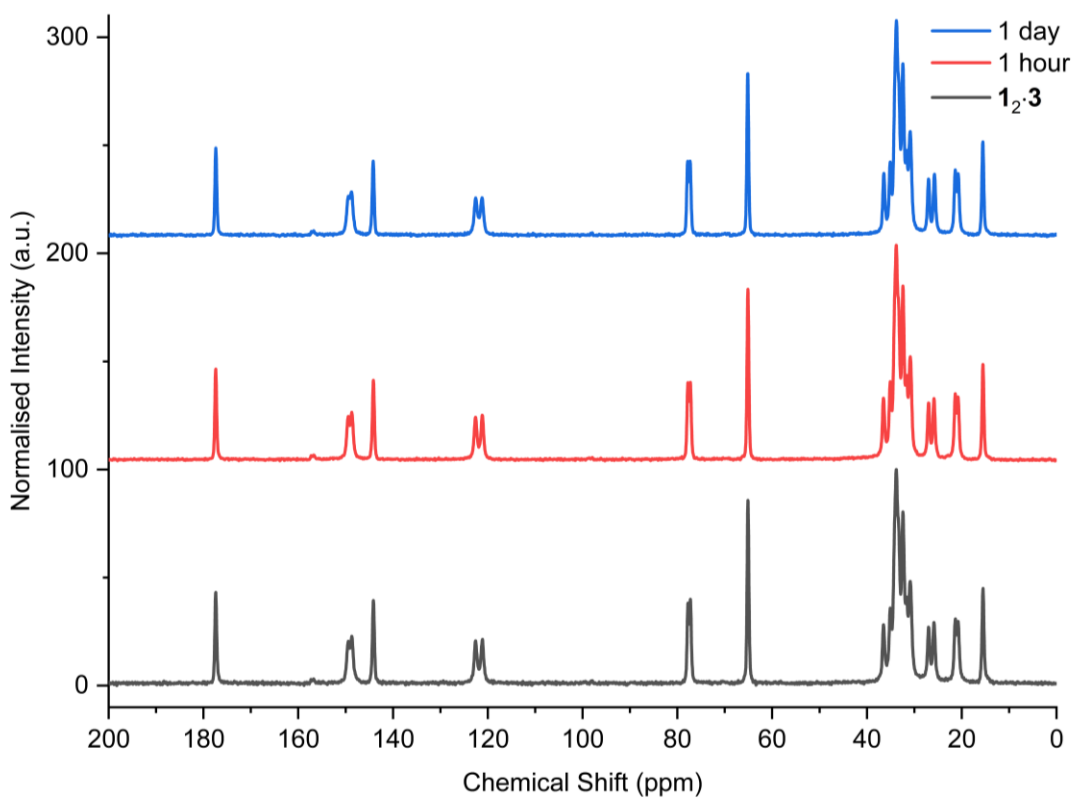


Figure S31. CP-MAS ^{13}C NMR spectra of $1_2\cdot 3$ irradiated for different durations by UV light at 254 nm.

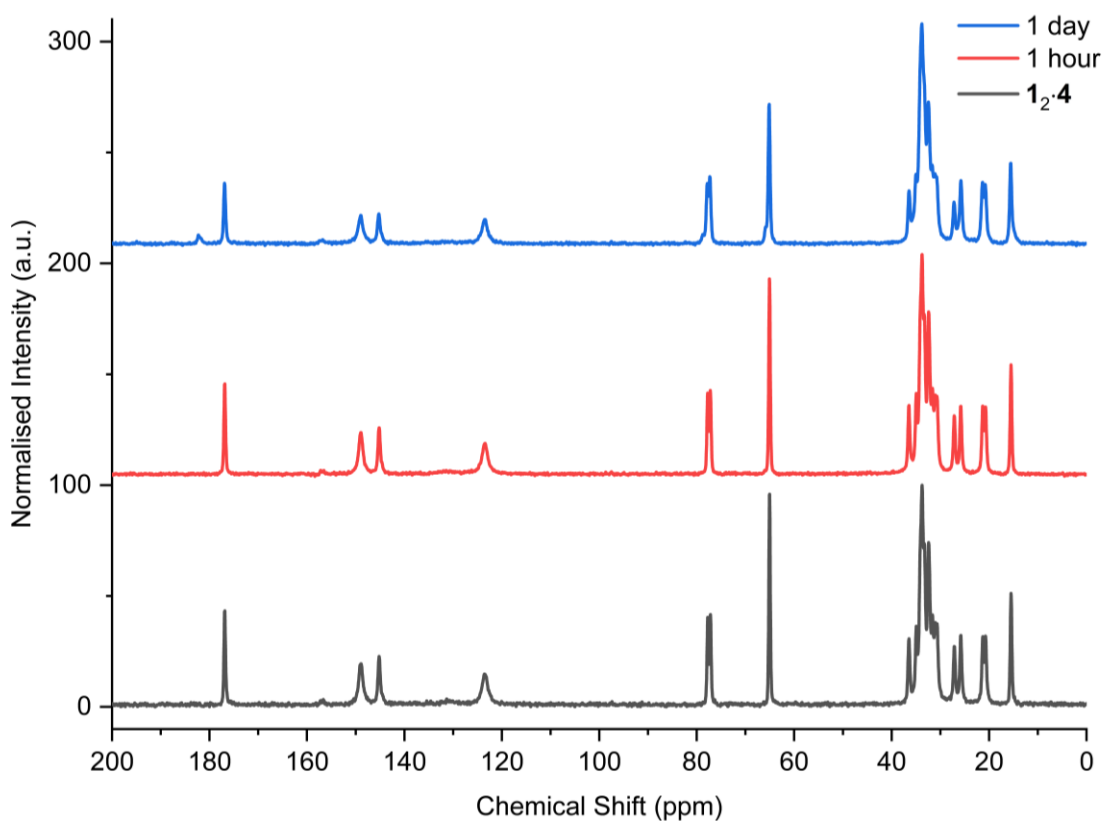


Figure S32. CP-MAS ^{13}C NMR spectra of $\mathbf{1_2 \cdot 4}$ irradiated for different durations by UV light at 254 nm. The additional peak at 180.3 ppm in the one-day irradiated spectra corresponds to the carboxylate peak of $\mathbf{1}$.

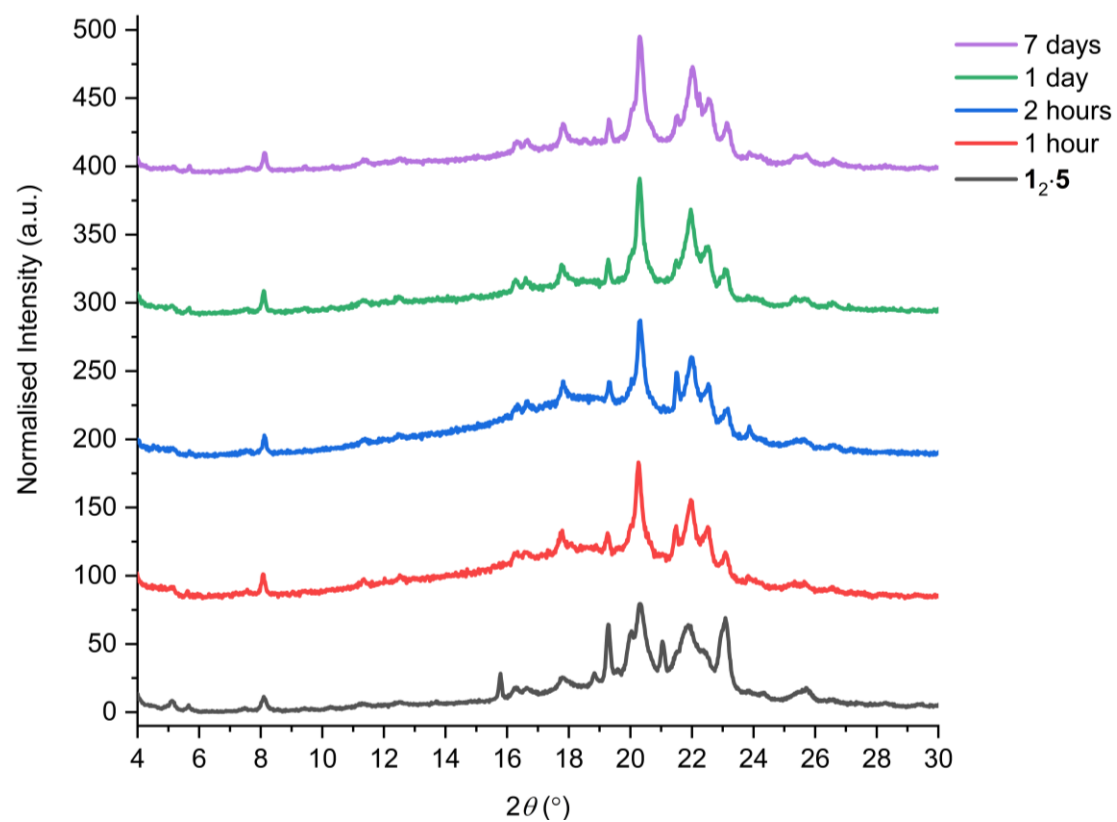


Figure S33. The experimental PXRD patterns of $1_2 \cdot 5$ irradiated for different durations by UV light at 254 nm.

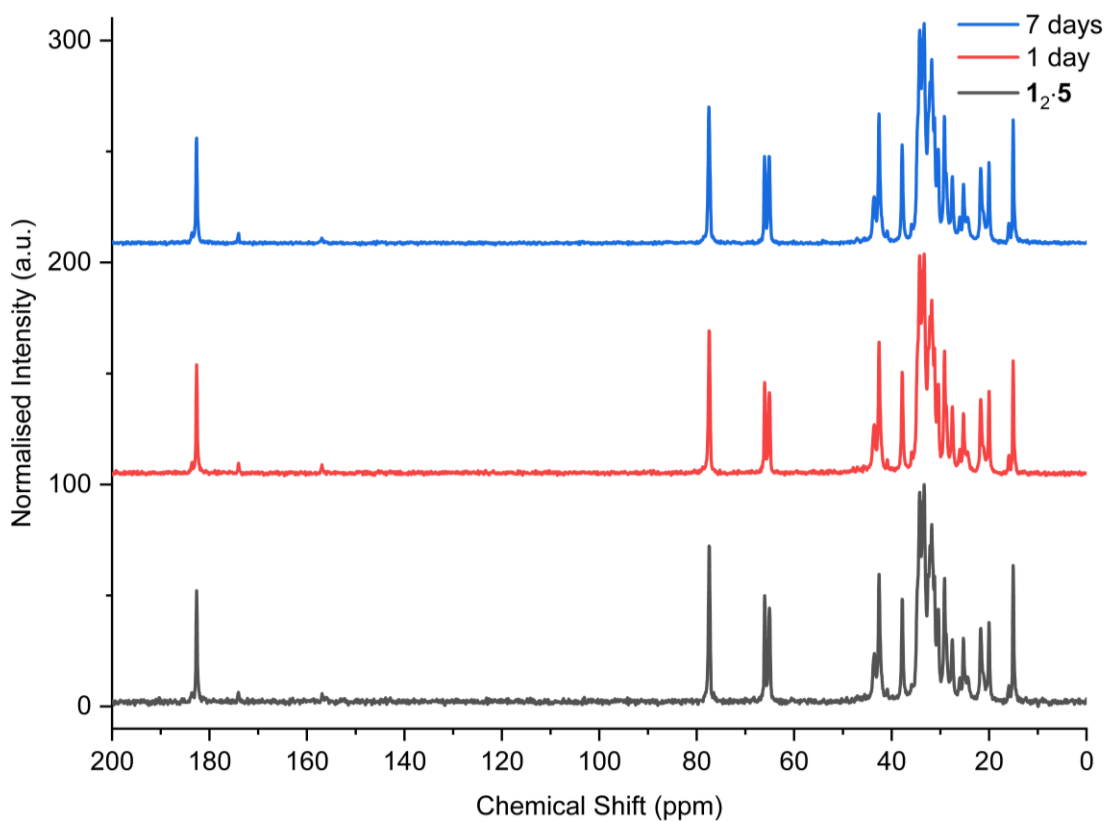


Figure S34. CP-MAS ^{13}C NMR spectra of 12.5 irradiated for different durations by UV light at 254 nm.

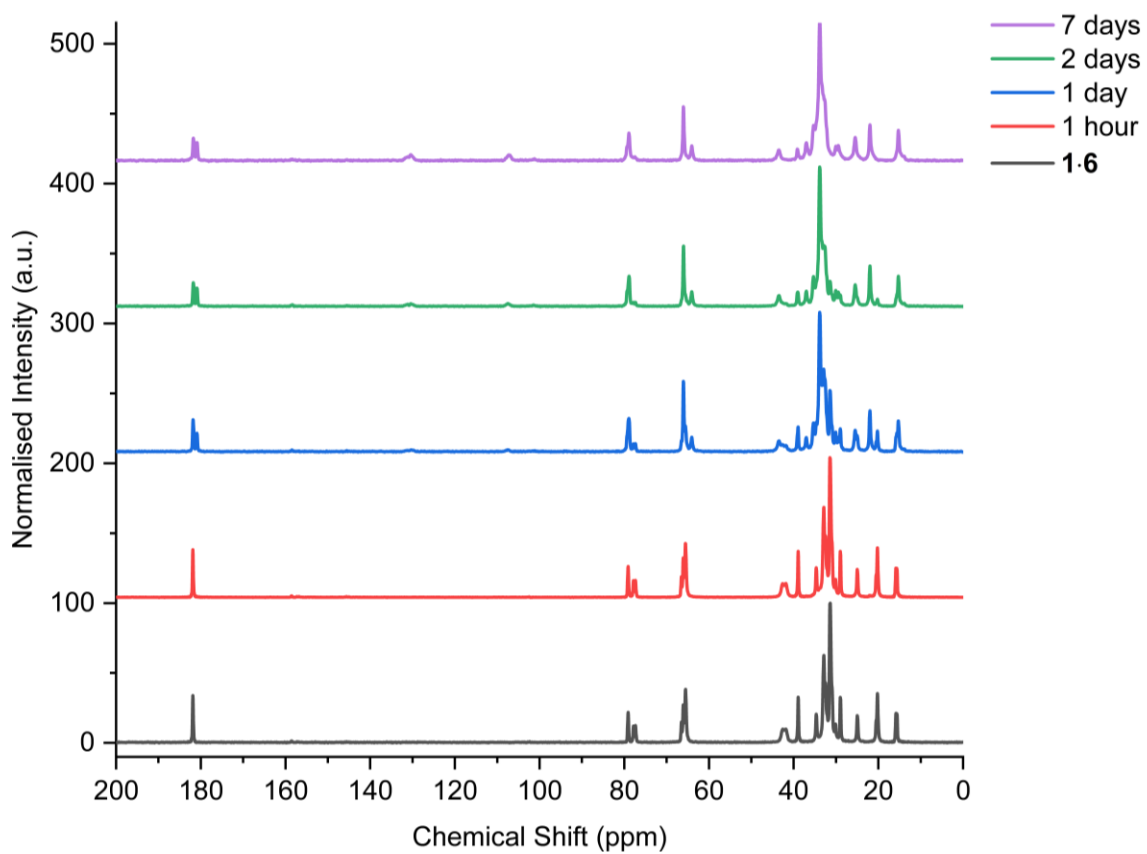


Figure S35. CP-MAS ^{13}C NMR spectra of **1-6** irradiated for different durations by UV light at 254 nm.

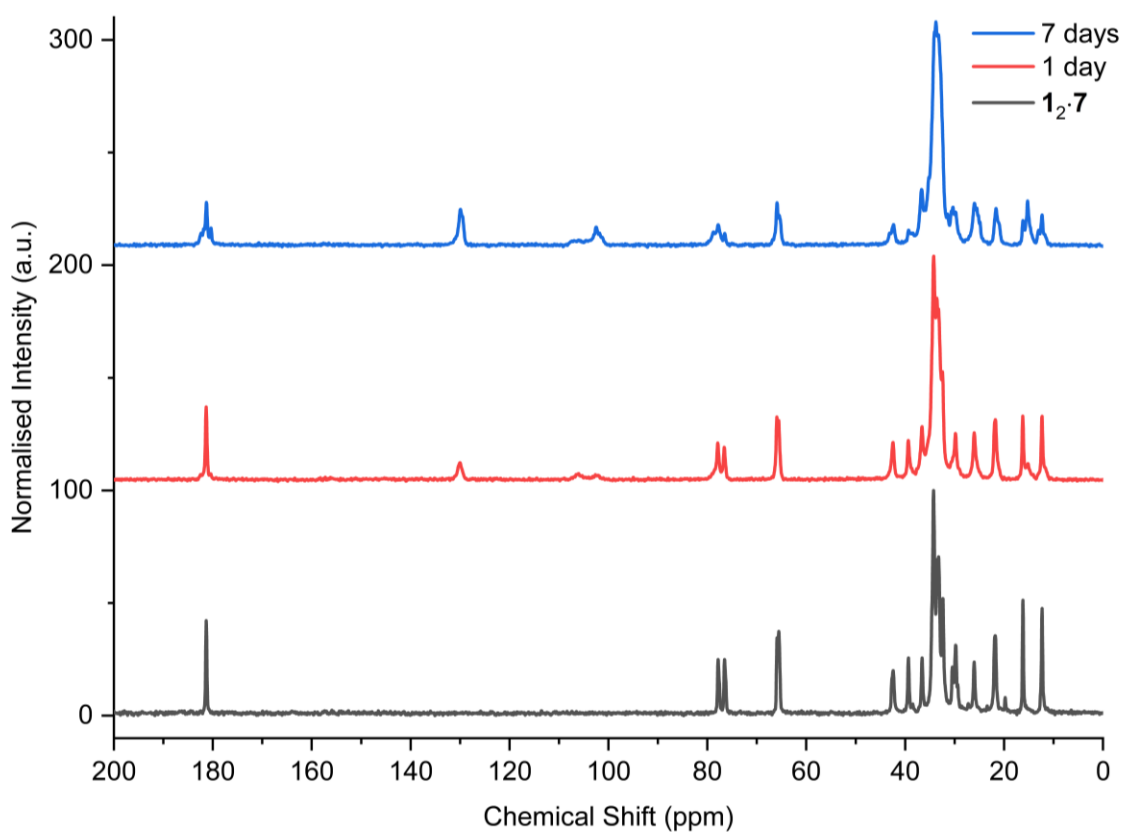


Figure S36. CP-MAS ^{13}C NMR spectra of $\mathbf{12\cdot7}$ irradiated for different durations by UV light at 254 nm.

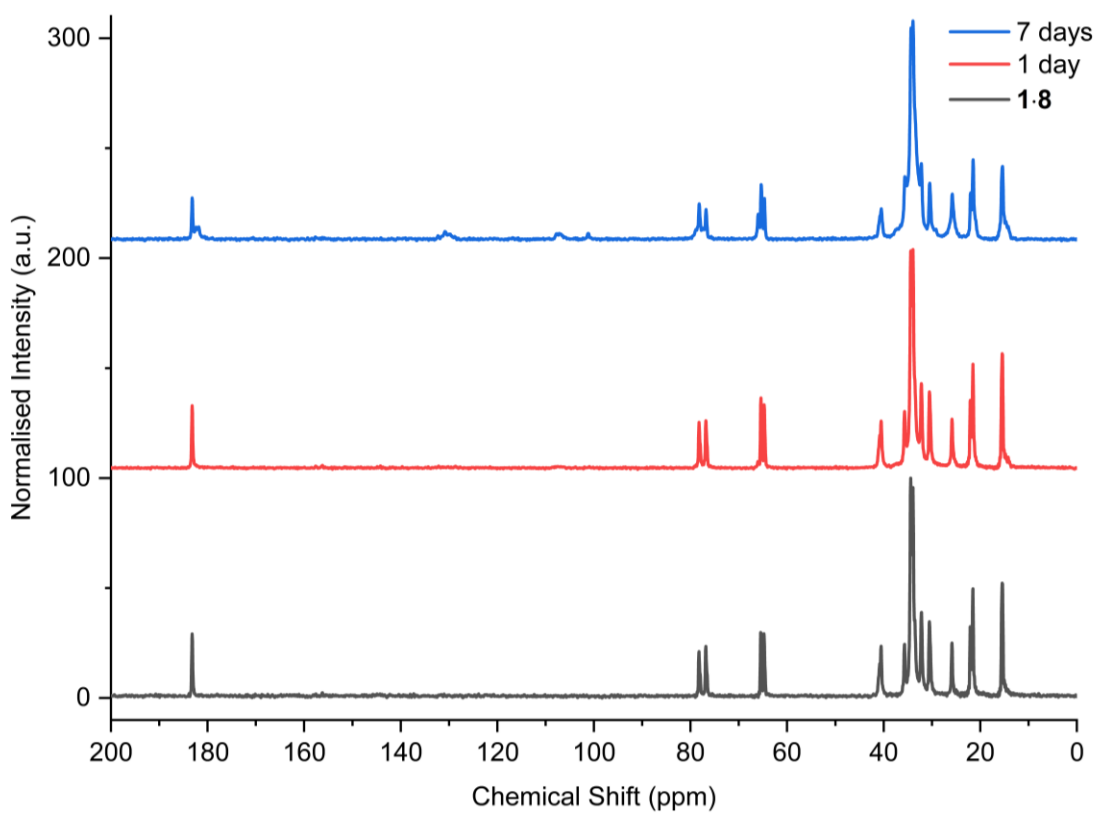


Figure S37. CP-MAS ^{13}C NMR spectra of **1-8** irradiated for different durations by UV light at 254 nm.

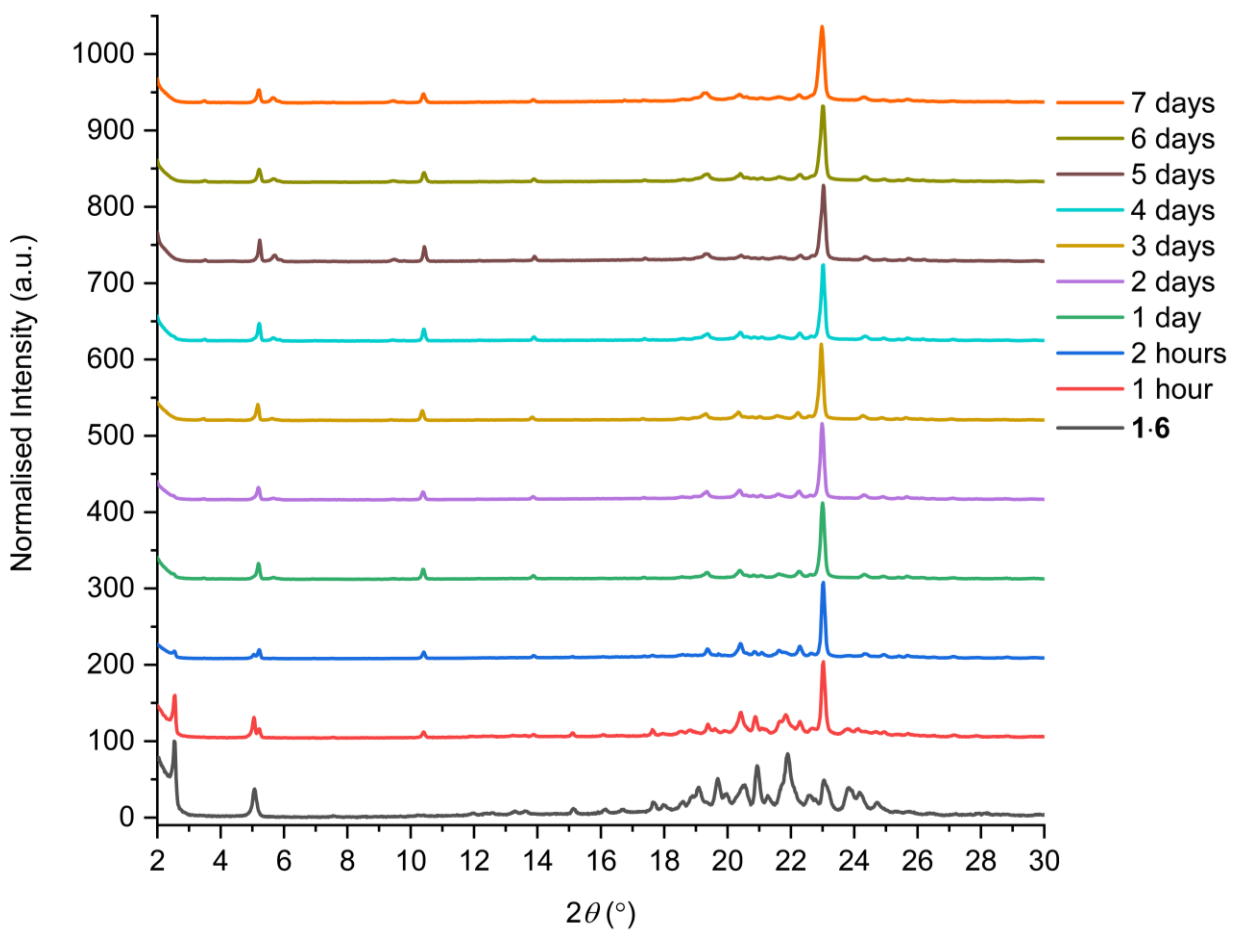


Figure S38. The experimental PXRD patterns of **1.6** irradiated for different durations by UV light at 254 nm.

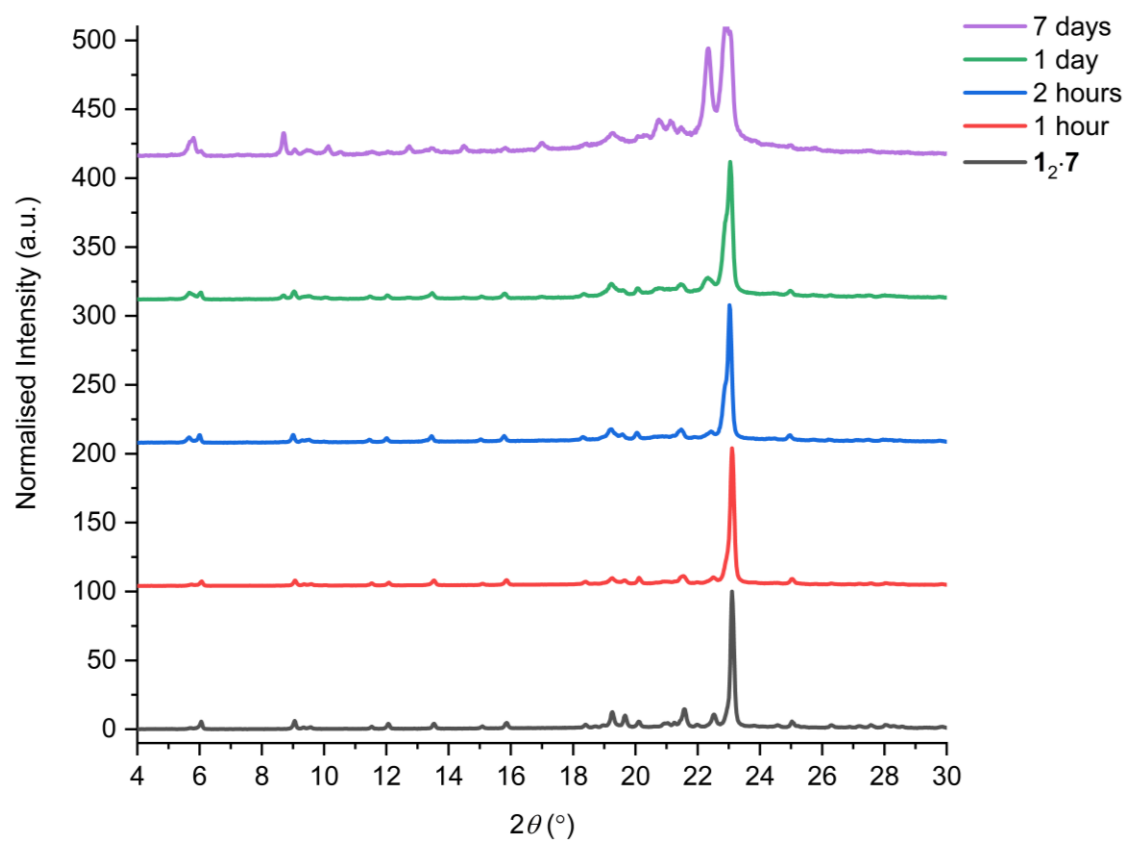


Figure S39. The experimental PXRD patterns of $1_2 \cdot 7$ irradiated for different durations by UV light at 254 nm.

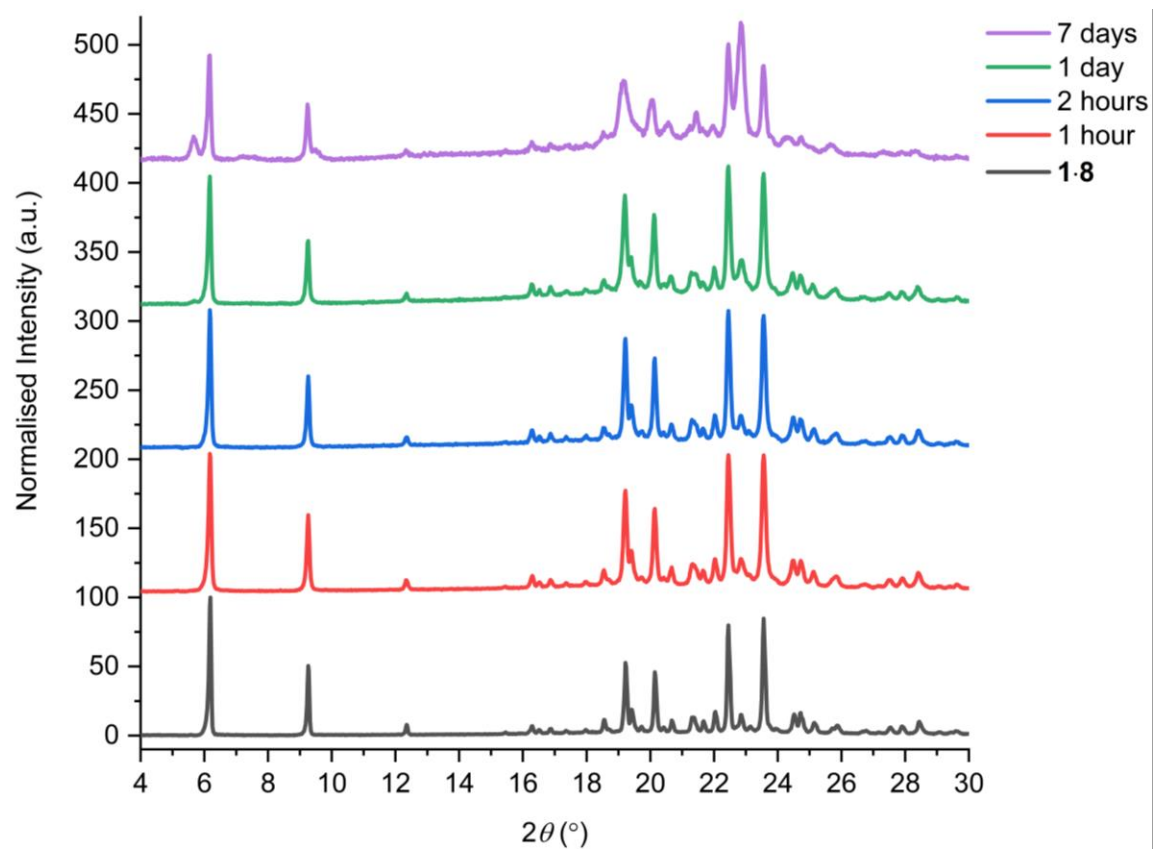


Figure S40. The experimental PXRD patterns of **1·8** irradiated for different durations by UV light at 254 nm.

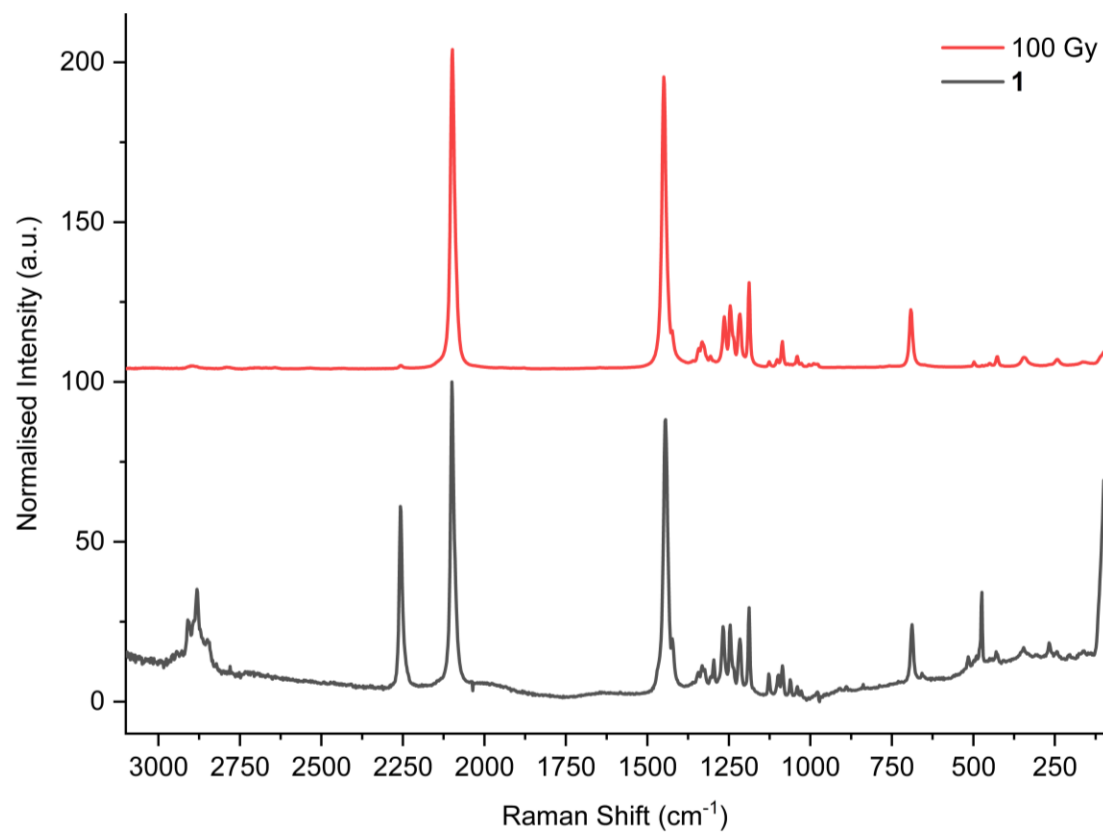


Figure S41. The Raman spectra of **1** before and after 100 Gy of X-ray radiation.

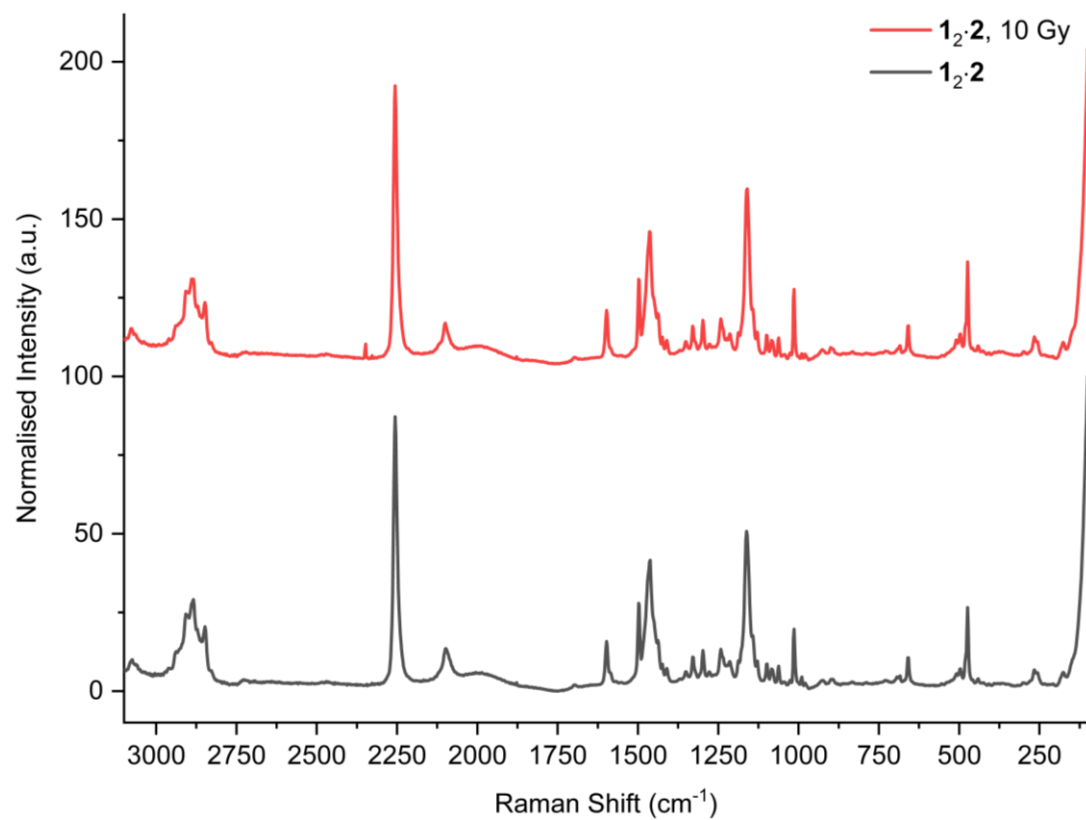


Figure S42. The Raman spectra of $1_2 \cdot 2$ before and after 10 Gy of X-ray radiation.

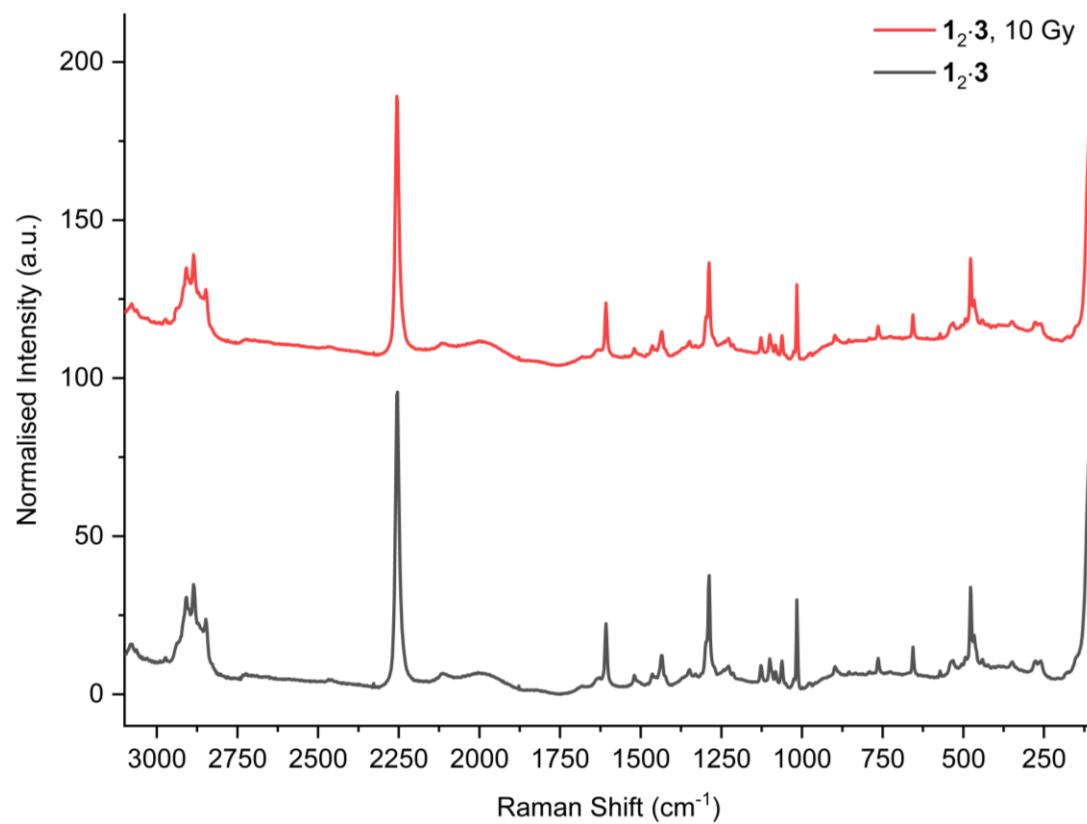


Figure S43. The Raman spectra of $1_2\cdot 3$ before and after 10 Gy of X-ray radiation.

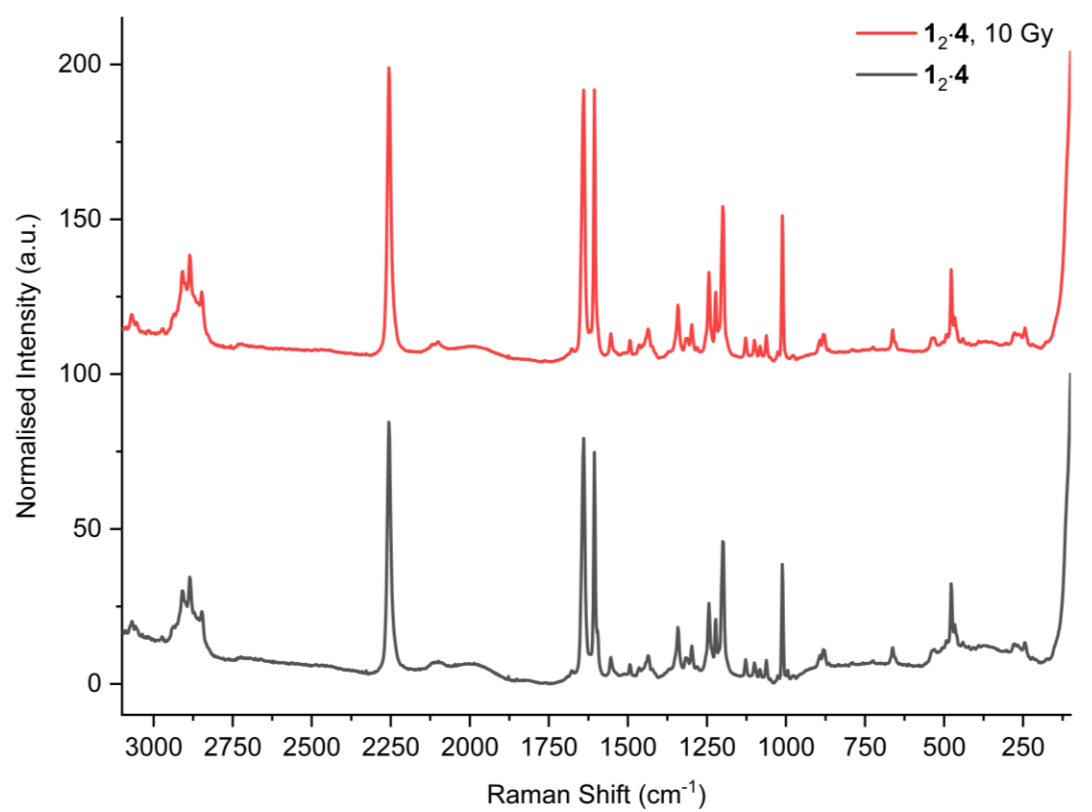


Figure S44. The Raman spectra of $1_2\cdot 4$ before and after 10 Gy of X-ray radiation.

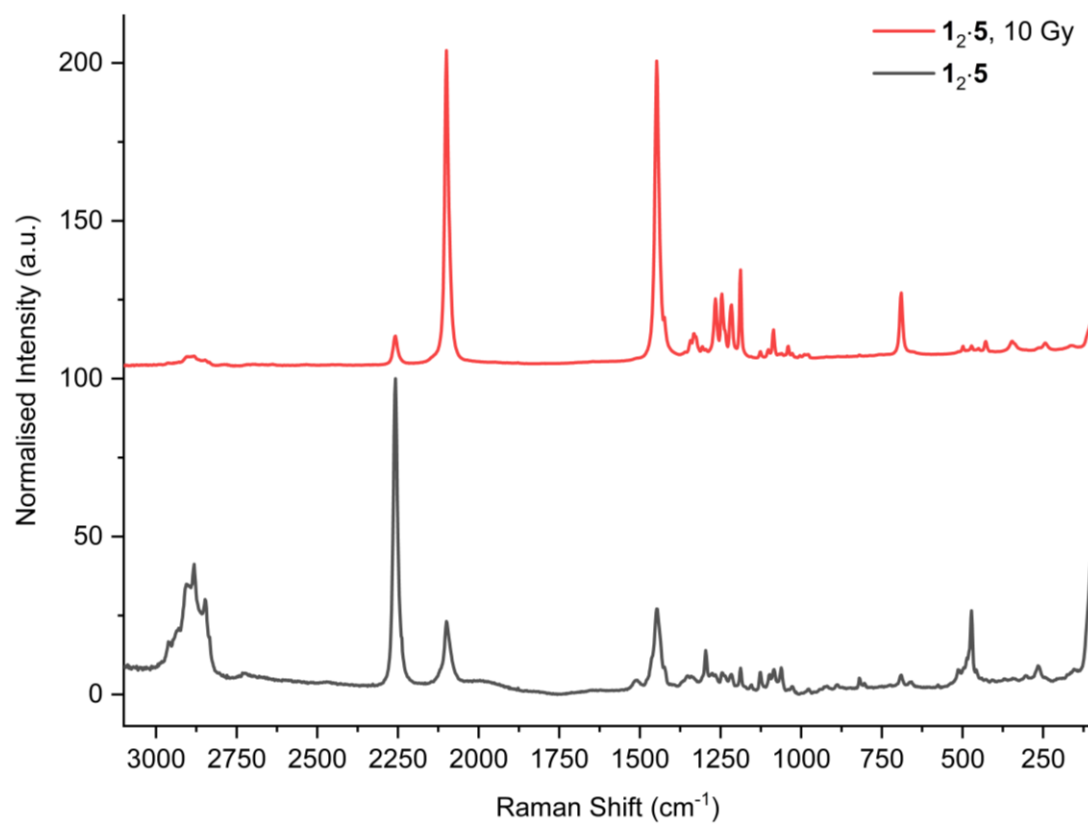


Figure S45. The Raman spectra of **1₂.5** before and after 100 Gy of X-ray radiation.

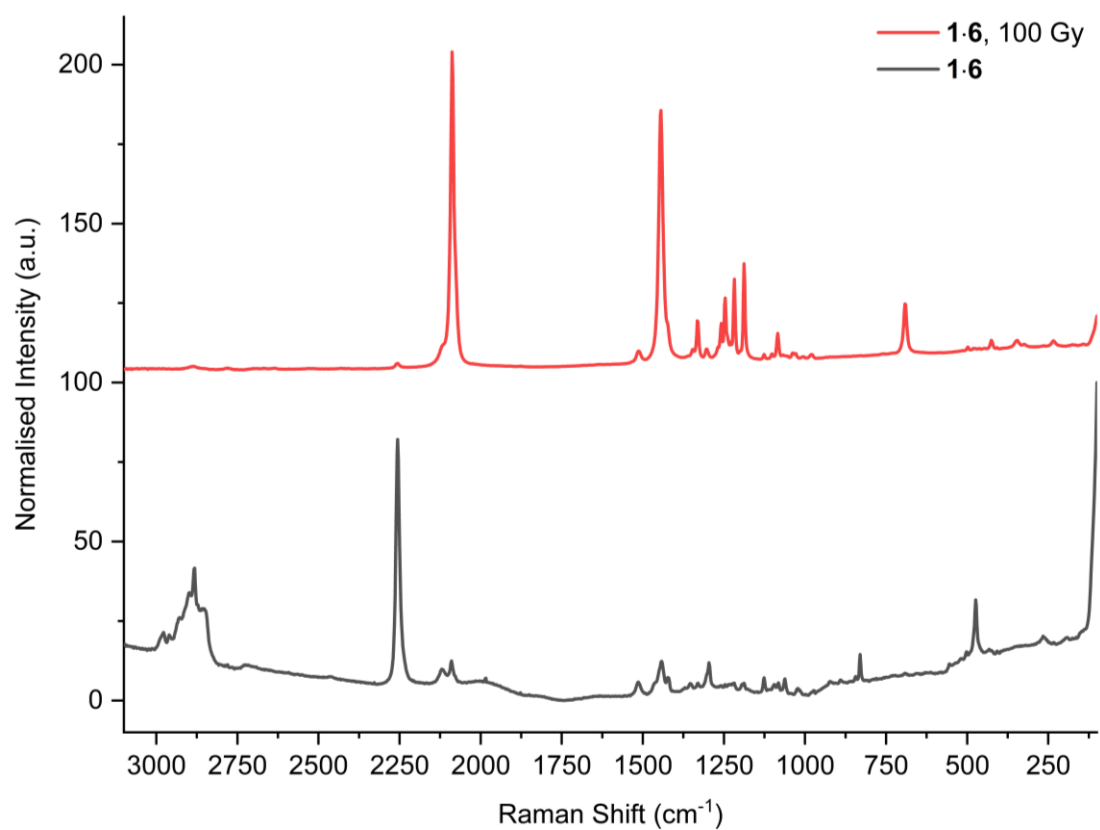


Figure S46. The Raman spectra of **1.6** before and after 100 Gy of X-ray radiation.

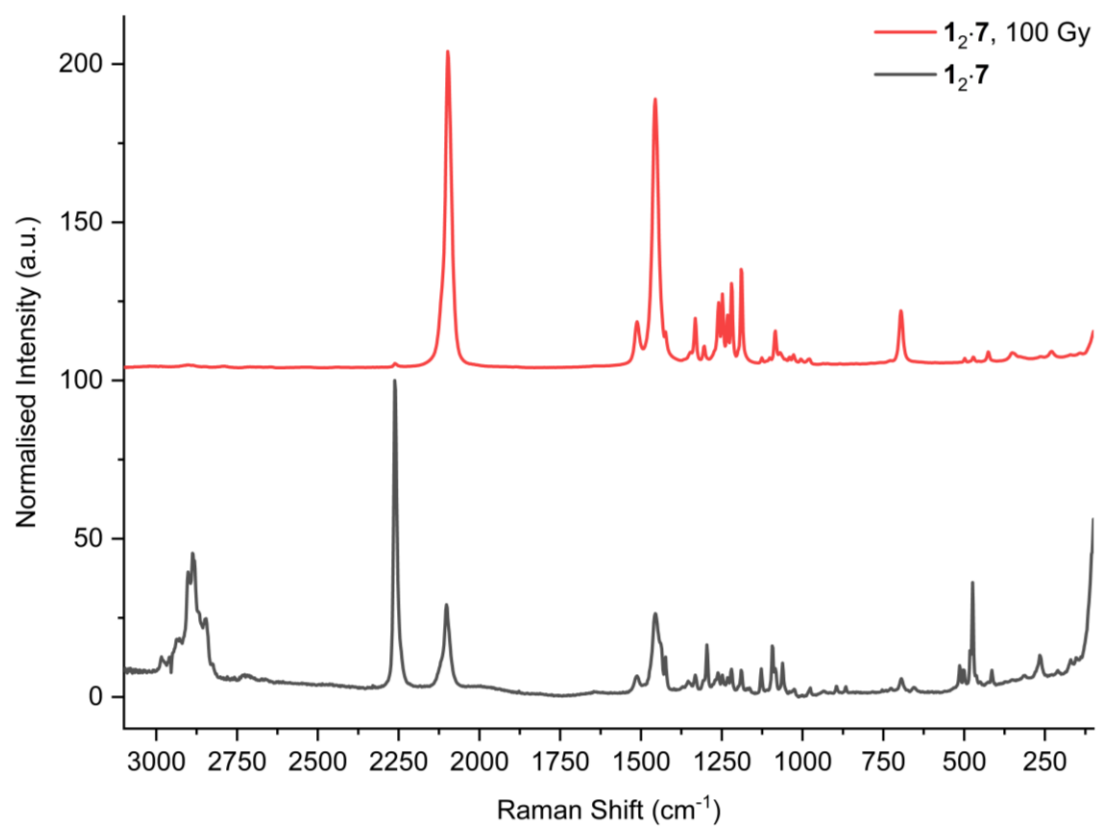


Figure S47. The Raman spectra of **1₂·7** before and after 100 Gy of X-ray radiation, highlighting the pre-resonance Raman effect by the differences in relative intensities of bands.

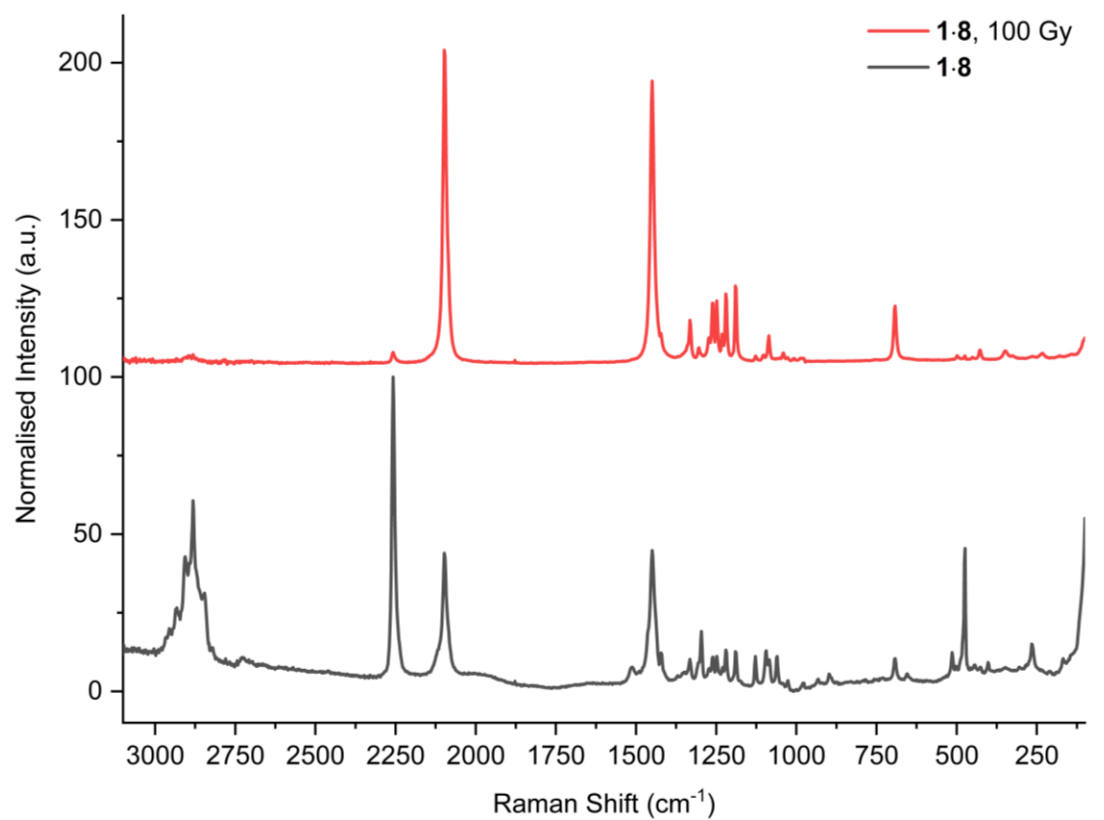


Figure S48. The Raman spectra of **1·8** before and after 100 Gy of X-ray radiation.